ANALYTICAL ABSTRACTS

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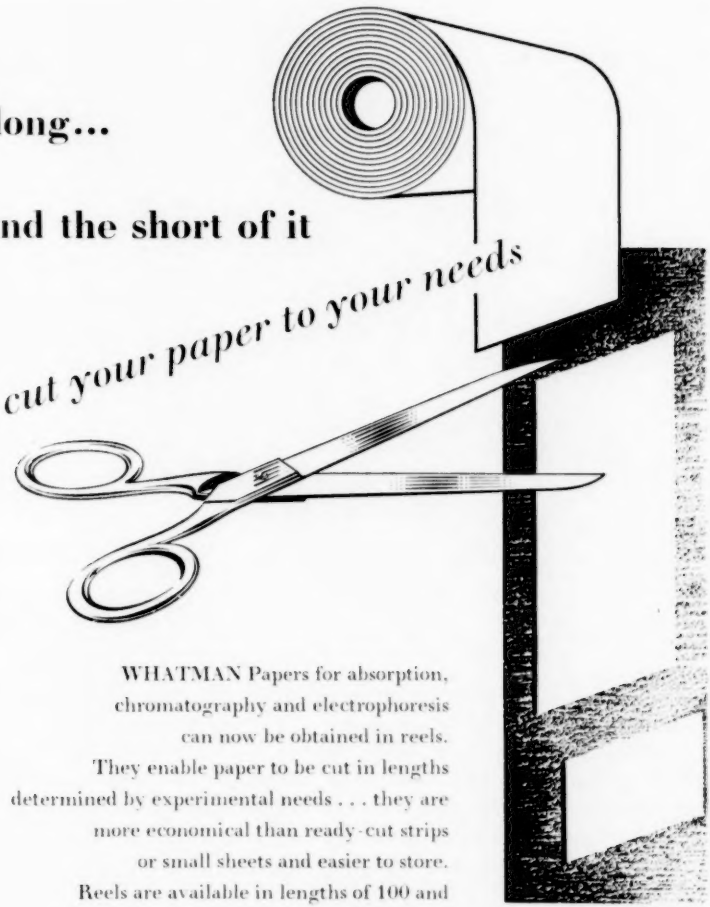

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1752. **Statistical evaluation of spectrographic methods.** B. N. Nelson (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *Appl. Spectroscopy*, 1957, **11** (3), 123–127.—The designing of experiments to give maximum information on all variables, with the lowest number of tests, in the development of an analytical method is discussed. The quality of the final method, of known precision and bias, when applied to routine analysis, is assessed by means of control samples. The results on these samples are plotted on control charts which show how day-to-day bias and precision are maintained. Worked examples are shown and a comprehensive bibliography is given.
P. T. BEALE

1753. **Analytical uses of pentacyanoferrate complexes.** E. F. G. Herington (Chem. Res. Lab., Teddington, England). *Ind. Chem. Mfr*, 1957, **33**, 439–440.—The reactions and analytical uses of $Na_2[Fe(CN)_5NH_3].H_2O$ and $Na_3[Fe(CN)_5NH_3].6H_2O$ are examined. Among the applications discussed are the use of the trisodium salt in the determination of *iso*quinoline in the presence of quinoline and quinaldine and as a spray reagent with dithiooxamide in paper chromatography.
S.C.I. ABSTR.

1754. **4-Semicarbazidoazobenzene and its use in analytical chemistry.** M. Winter, E. Demole and E. Sundt (Firmenich & Cie., Geneva). *Helv. Chim. Acta*, 1957, **40**, 467–476.—The preparation of this compound and the properties of its cryst. derivatives with a number of aldehydes and ketones are described. It may be used in column or paper chromatography to isolate or identify aldehydes and ketones in fruits. N. E.

1755. **Multi-purpose standard for microchemical analysis.** W. H. Smith (Nat. Bureau of Standards, Washington, D.C., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 149–150.—5-Chloro-4-hydroxy-3-methoxybenzylthiuronium phosphate is suggested for use in the ultimate analysis of organic compounds.
K. A. PROCTOR

1756. **Chelatometry. XV. New pM indicators.** P. Wehber (Lab. Metallhütte, Mark A.-G., Hamburg-Wilhelmsburg, Germany). *Z. anal. Chem.*, 1957, **158** (1), 10–14.—Recent applications of the pM indicators 4-(2-pyridylazo)resorcinol (PAR), murexide, ninhydrin and Eriochrome red B are discussed. (24 references.) A. R. ROGERS

1757. **Fast grey [4-(2-hydroxy-3-nitro-5-sulphophenylazo)-2-naphthol] as a new indicator in titrations with EDTA.** H. Khalifa (2nd Chem. Inst., Univ. Vienna). *Anal. Chim. Acta*, 1957, **17** (2), 194–198 (in English).—A 0·1% aq. soln. of Fast grey RA is a suitable indicator for the determination of Th, Fe^{III}, Bi or Zn at pH $\simeq$2. Interference in the determination of Th arises from the presence of Fe^{III}, Bi and Zr; up to 25% of U and excess of rare-earth metals do not interfere, nor do Ca, Ba, Mg, Be or Zn. The determination of Th in the presence of Fe is possible if ascorbic acid is added. Rare-earth metals, Bi, Th and Zn interfere in the determination of Fe, as do Fe^{III}, Pb and Cu in the determination of Bi, and Fe^{III} and Bi in the determination of Zn. Details are given for the analysis of mixtures of Fe and Th, Zn and Ca, Zn and Mg, and Zn and Th. J. H. WATON

1758. **Analysis for industry [analytical applications of chemiluminescence].** L. Erdey (Tech. Univ., Budapest). *Ind. Chem. Mfr*, 1957, **33**, 459–461.—The use of chemiluminescent substances as indicators for determining titration end-points in turbid or coloured solutions is discussed, with particular reference to the use of Lucigenin (5:5′-diacridyl dimethonitrate). S.C.I. ABSTR.

1759. **Principles of absorptiometric iodimetry.** C. Herbo and J. Sigalla (Fac. des Sci., Univ. Libre, Brussels). *Anal. Chim. Acta*, 1957, **17** (2), 199–207 (in French).—Oxidising agents and reducing agents can be determined absorptiometrically by their influence on the I_3^- spectrum. All the iodine must be present as I_3^-, and that amount of iodine produced by the autoxidation of I^- must be negligible. The limit of the method is 2×10^{-7} g-equiv. of oxidising or reducing agent per litre, when a 5-mm cell is used. J. H. WATON

1760. **Studies of bivalent chromium salts. I. Standardisation of chromous solutions with ferric and cupric salts, using visual indicators.** J. P. Tandon and R. C. Mehrotra (Chem. Lab., B.R. Coll., Agra Univ., India). *Z. anal. Chem.*, 1957, **158** (1), 20–24 (in English).—Methylene blue, indigo carmine, neutral red, methyl red and phenosafranine serve as excellent internal indicators for the titration of Fe^{3+} with $CrSO_4$ soln. Phenosafranine and neutral red are suitable internal indicators for the titration of Cu^{2+} with $CrSO_4$ soln. in the presence of a high concn. of HCl. The concn. of a $CrSO_4$ soln. calculated from titrations of Fe^{3+} and Cu^{2+} soln. agree within experimental error ($\pm$0·5%). A. R. ROGERS

1761. **Potentiometric electrode systems in non-aqueous titrimetry.** J. T. Stock and W. C. Purdy (Univ. Connecticut, Storrs, U.S.A.). *Chem. Rev.*, 1957, **57** (6), 1159–1177.—A review with 231 references. N. E.

1762. **Potentiometric titration of very weak acids. Influence of titrant solvent.** G. A. Harlow and G. E. A. Wyld (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 73.—The

inflections obtained from the potentiometric titration of very weak acids, such as phenols, in acetone and pyridine solvents are greatly affected by the acidity of the titrant solvent. Tetra-*n*-butylammonium titrants prepared in *iso*propyl alcohol are superior to titrants prepared in ethanol, methanol or water. Methanol exhibits the same harmful effect as water in titrations of weak acids in non-aq. solvents. G. P. COOK

1763. **The production of a continuously variable liquid mixture for gradient elution in chromatography and similar applications.** E. Geiss (Bundesforschungsanstalt f. Viruskrankheiten der Tiere, Tübingen). *Hoppe-Seyl. Z.*, 1957, **308** (2–4), 74–80.—The methods available for the production of liquid mixtures of variable composition are surveyed and a table is given showing the concn. curves obtained with the various types of apparatus. For experiments with ion-exchange resins, a mixing apparatus was sought capable of yielding a concn. curve which, from an initial horizontal value, should pass through an exponential increase to a linear value. Details are given of the construction, based on mathematical considerations, of such an apparatus. Evidence is presented that such curves are obtained.
F. POWELL

1764. **Exploratory studies of high-temperature gas - liquid chromatography.** J. M. Ogilvie, M. C. Simmons and G. P. Hinds, jun. (Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 25–27.—A gas - liquid chromatography system, which is capable of operating up to $\simeq$400°, is described. The preheater, separation column and detector are all cast in an aluminium block and are heated electrically. The stationary liquids employed are the thermally stable, and sufficiently non-volatile, petroleum-derived asphaltenes. This procedure was successfully applied to the determination of normal paraffin distribution in waxes, results being generally within 10% of those obtained by a mass-spectrometric method. G. P. COOK

1765. **An adsorption theory of gas chromatography.** L. Gráf and J. Tóth (Natural Oil Mining Service, Nagykanizsa, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1958, **13** (3–4), 403–427.—A theory of elution and frontal gas chromatography is developed. An equation is given for the correlation between the two methods. The conditions determining the possibility of separation of two gases by elution are expressed mathematically.
J. S. HETMAN

1766. **Coulometry applied to gas-phase chromatography.** A. Liberti (Ist. di Chim. Anal., Univ. Messina, Italy). *Anal. Chim. Acta*, 1957, **17** (2), 247–253 (in English).—Coulometric methods are readily applicable to the determination of gases in vapour-phase chromatography. The eluted gases are passed into a cell containing an absorbing electrolyte and are titrated if they possess some suitable functional group. Photometric, potentiometric and amperometric end-points are employed. In this way, carboxylic acids, bases, thiols, aldehydes and ketones may be determined. If the gases are burnt after leaving the column, then any volatile organic compound can be estimated as CO_2. J. H. WATON

1767. **Ion exchange.** J. Schubert (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. TID–7512, 1956, 183–192.—A review and discussion are presented with special attention to application of ion-exchange separation techniques to agricultural problems. General applications, choice of resins, feasibility of separation procedures, and specific analytical methods are discussed. In many of the methods, radioactive tracers are used. (16 references.) CHEM. ABSTR.

1768. **The application of radioactive isotopes for developing new methods in analytical chemistry. Organic co-precipitants.** V. I. Kuznetsov (Inst. for Geochem. and Anal. Chem., Acad. Sci., Moscow). UNESCO/NS/RIC/46, 1957, 17 pp. (in English).—Three classes of method are considered—(*a*) co-pptn. of elements as normal salts, (*b*) co-pptn. of elements as uncharged complex compounds, (*c*) co-pptn. by a colloid chemical mechanism. Many elements may be co-pptd. by the first two methods; the third method is particularly suitable for the co-pptn. of Nb, Ta, W, Mo and Zr. G. J. HUNTER

1769. **The use of radioactive isotopes in spectral analysis.** E. E. Weinstein, L. I. Pavlenko and Y. I. Belyaev (V. I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. Sci., Moscow). UNESCO/NS/RIC/47, 1957, 23 pp. (in English).—The use of radioactive tracers has enabled the relationship between the intensity of a spectral line and the evaporation of an element to be studied; results are given for the evaporation of La, Mo, Sb and Na from graphite and diorite. The determination of Mo in various silicate rocks has been investigated and an improved procedure is given. Optimum conditions have been established for the evaporation and collection of traces of Cd, Sn, Sb, Pb and Bi from high-purity WO_3. Results are given for the condensation of Sb and Tl at various temperatures after evaporation from the oxides of Zr, La, Be and U. Conditions are established that render results independent of the method of preparation of standards and the chemical nature of the matrix.
G. J. HUNTER

1770. **Wet oxidation of organic matter employing perchloric acid at graded oxidation potentials and controlled temperatures.** G. F. Smith (Univ. Illinois, Urbana, U.S.A.). *Anal. Chim. Acta*, 1957, **17** (2), 175–185 (in English).—Graded potential oxidation, in which controlled concn. of $HClO_4$ with or without the addition of H_2SO_4 or HNO_3 is used, permits the controlled wet-ashing of organic matter. The apparatus used is a modified form of that described by Bethge (*cf. Anal. Abstr.*, 1954, **1**, 1727).
J. H. WATON

2.—INORGANIC ANALYSIS

1771. **Infra-red spectra of oxine chelates.** J. P. Phillips and J. F. Deye (Dept. of Chem., Univ. of Louisville, Ky., U.S.A.). *Anal. Chim. Acta*, 1957, **17** (2), 231–233 (in English).—The i.r. spectra of 33 metal oxinates, in KBr pellets, have been examined over the range 2 to 15 μ. Some metal oxinates can be identified by comparison of wavelength and intensity of their absorption bands, and by comparing the density of the band due to water of hydration at 3 μ with that of the band at 9 μ, it is possible to determine the no. of mol. of water of hydration in the chelate. J. H. WATON

1772. **Disubstituted dithiocarbamates. VI. Displacement reactions between metal diethyldithiocarbamates in organic phase and metal ions in aqueous phase.** H. Bode and K.-J. Tusche (Anorg.-chem. Inst., Tech. Hochsch., Hannover, Germany). *Z. anal. Chem.*, 1957, **157** (6), 414–427 (in German).—Displacement reactions between metal diethyldithiocarbamates dissolved in CCl_4 and metal ions in aq. phase have been quant. investigated for Hg^{2+}, Pd^{2+}, Ag^{+}, Cu^{2+}, Tl^{3+}, Ni^{2+}, Bi^{3+}, Pb^{2+}, Co^{3+}, Co^{2+}, Cd^{2+}, Tl^{+}, Zn^{2+}, In^{3+}, Sb^{3+}, Fe^{3+}, Te^{4+}, Mn^{2+} and Mn^{3+} at pH 8·5 and pH 11; no masking agent is employed. Even when the displacement is only partial, a large excess of the cation in the aq. phase may produce quant. displacement.

J. H. WATON

1773. **Separation and determination of Cu^{2+}–Ni^{2+} and Ni^{2+}–CrO_4^{2-} by electrochromatography.** L. Cavallaro, C. Bighi and G. Trabanelli (Ist. Chim., Univ. Ferrara, Italy). *Ann. Chim., Roma*, 1957, **47** (2), 189–194.—Experiments in the use of electrochromatography for the separation of various pairs of ions are described. Details are given of the solvents and currents used; the principal defect is claimed to lie in the colorimetric method used for the determination of the ions, but so far no better system has been found. J. F. P. H. GREENE

1774. **Application of continuous electrochromatography to the separation of certain pairs of characteristic anions and cations.** C. Bighi and G. Trabanelli (Ist. Chim., Univ. Ferrara, Italy). *Ann. Chim., Roma*, 1957, **47** (2), 195–202.—The work reported in a previous paper (*cf.* Cavallaro *et al.*, *Anal. Abstr.*, 1958, **5**, 1773) has been extended and used for the separation of several other pairs of ions, in particular to the separation of the complexes of Fe with 2:2′-dipyridyl. The behaviour of polyionic anions was also studied. Full details of the experimental methods and of the results are given, with illustrations of several typical chromatograms.

J. F. P. H. GREENE

1775. **Applications of partition chromatography to the determination of certain elements in minerals. II. Detection and semi-quantitative estimation of silver, nickel, cobalt, copper, niobium, tantalum and titanium.** H. Agrinier (Lab. de Min. du C.E.A., Châtillon, Seine). *Bull. Soc. Franç. Minér. et Crist.*, 1957, **80**, 275–292.—Details are given for the detection and estimation of these metals in minerals by comparison of the colours obtained with those given by standards, the techniques previously outlined (*Anal. Abstr.*, 1958, **5**, 358) being used.

R. A. HOWIE

1776. **Determination of the impurity concentrations in a semi-conductor from Hall coefficient measurements.** P. A. Lee (British Thomson-Houston Co. Ltd., Rugby, England). *Brit. J. Appl. Phys.*, 1957, **8** (8), 340–343.—An equation is derived relating carrier concn. (p) with temp. (T), acceptor concn. (N_A) and donor concn. (N_D) in a semi-conductor. The curve of p *vs.* $1/T$ can be obtained by measuring the Hall coeff. (R) over a range of temp., whence the equation and this curve can be used to calculate N_A and N_D. The procedure for calculating the limits of determination of impurities, *e.g.*, B, in p-type Si is shown. For this element the concn. (N_A) of B may be as low as 10^{13} atoms per cu. cm, whilst R is now known down to 20° K.

W. J. BAKER

1777. **Determination of low concentrations of deuterium in water with a mass spectrometer. [I.] The exchange equilibrium method.** Atsushi Mizuike (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *Japan Analyst*, 1957, **6** (6), 383–386.—The water containing a low concn. (< 1 atom %) of deuterium is equilibrated with H at 25° and the HD produced is introduced into a mass spectrometer for the measurement of its deuterium content. The equilibrium between sample water (5 ml) and H (1 atom, ≃25 ml) is achieved within 3 hr. in the presence of Adam's platinum oxide catalyst (10 mg) and fumaric acid (5 mg).

[**II.**] **The zinc decomposition method.** Atsushi Mizuike. *Ibid.*, 1957, **6** (6), 386–387.—Attempts were made to minimise the "memory" effect of Graff and Rittenberg's apparatus (*Anal. Chem.*, 1952, **24**, 878) for the decomposition of water with granulated zinc. A simple apparatus is proposed. Fresh granulated zinc (30 mesh, 30 g) is used for each measurement; the adsorbed water is removed by heating *in vacuo*. The results are slightly lower (by 2 to 3% relative) than those by the exchange equilibrium method. K. SAITO

1778. **Determination of the tritium content of liquids.** E. G. Gracheva and Sh. G. Khusainova. *Atomnaya Energiya*, 1957, **2** (1), 70; *Ref. Zhur., Khim.*, 1957, Abstr. No. 51,605.—In determining tritium in liquids not containing other radioactive elements, it is proposed to use the weak radiation evoked in the substance of the sample by the β-particles of the tritium. Place the liquid in a thin layer (depth 1·5 cm) in front of a tube of the end-window type (window 4 mg per sq. cm, diam. ≃40 mm); cover the vessel with an aluminium lid (thickness 18 μ). The quantity of tritium in the sample is found by comparison with a standard. The described method determines $\leqslant 10^{-4}$ curies of tritium per litre. C. D. KOPKIN

1779. **Volumetric determination of moisture in gases.** F. M. Rapoport. *Trudy Gos. Nauch. i Proektn. Inst. Azotn. Prom.*, 1954, (3), 214–218; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,830.—Pass the gas through two absorbers in series containing 5 ml each of ethanediol or pyridine, and titrate the soln. in the absorbers with Karl Fischer reagent. The rate of passage of gas is 12 litres per hour. Obtain a blank by titrating 5 ml of ethanediol or pyridine under analogous conditions. The relative error is 2% with a moisture content in the gas of $\leqslant$50 mg per cu. metre. The analytical results agree well with those obtained by the gravimetric method. C. D. KOPKIN

1780. **Polarographic determination of hydrogen peroxide and aliphatic acids in mixtures.** B. B. Radak and B. L. Djukanović (Inst. Nucl. Sci. "Boris Kidrich," Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 59–63 (in English).—The critical concn. ratio ($C_{H_2O_2}/C_{Acid}$) above which it is impossible to determine the acid has been determined for formic acid (0·62), acetic acid (0·44), oxalic acid (1·2), succinic acid (0·79) and citric acid (1·55). Hydrogen peroxide can be determined in a mixture with oxalic acid only when $C_{H_2O_2}/C_{H_2C_2O_4}$ is >0·05, in 0·1 *N* Li_2SO_4. In the instances when the acid is present in large excess, the H_2O_2 can be determined after adjusting the pH to 5 with KOH soln.; the supporting electrolyte used is 0·1 *N* Li_2SO_4 mixed with K oxalate, formed in solution.

J. H. W. FORSYTHE

1781. **The spectrographic analysis of high-purity lithium compounds.** R. V. Jury (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1871, 1957, 12 pp.—The lithium salt (as sulphate) is dried, fused at 900° and finely ground; a charge of 100 mg is then mixed with an equal wt. of graphite and is completely burnt in a deep graphite cup in a d.c. arc (15 amp.), background being minimised by masking the hot electrode-tips. A Hilger 3-metre grating spectrograph combined with a short-focus lens is used. The spectra are compared with those of standards containing added elements. The limits of detection (p.p.m.) are less than 1 (for Mo, Mn and V), 1 to 2 (for Cr, Ni, Fe and W), 5 (for Zn), and 10 (for Ta and Nb). W. J. BAKER

1782. **Determination of radio-caesium by complex cobalticyanide precipitation.** J. C. Langford (Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. HW-49,668, 1957, 30 pp.—Cobaltous cobalticyanide was found to be a most satisfactory precipitant for radio-caesium because of its stability in strongly acid soln. and its freedom from other radio-contaminants in such a soln. A procedure has been developed which gives a decontamination factor $>10^5$ for the gamma-emitting contaminants, Zr - ^{95}Nb, ^{106}Ru, ^{144}Ce and ^{125}Sb. The recovery of radio-caesium is >99%. The only substances found to interfere with quant. separation in 3 *N* H_2SO_4 were NH_4^+ at >0·5 *M* concn. and HNO_3 at >*M* concn. After dissolving the ppt. a volatilisation of Ru is carried out in ≃5 *N* H_2SO_4 made ≃0·2 *M* with HIO_4. The excess of HIO_4 is removed by decomposition in boiling H_2SO_4 soln. Further decontamination is achieved by a $Fe(OH)_3$ scavenging and a re-pptn. of the cobaltous cobalticyanide from acid soln. N. E.

1783. **Determination of Cu^+ in the presence of Cu^{2+}, H^+ and NH_4^+ and various unsaturated organic substances for the control of solutions of catalysts in the manufacture of vinylacetylene by dimerisation of acetylene.** O. Frehden and I. Conu. *Rev. Chim., Bucharest*, 1957, **8** (9), 600–604.—The principle of this method is an oxidation - reduction whereby Fe^{3+} are reduced to Fe^{2+} and Cu^+ are oxidised to Cu^{2+}. The soln. to be determined is titrated directly with a standard soln. of Fe^{3+} (iron alum) in an atmosphere of methane, with sulphosalicylic acid as internal indicator. The method is claimed to be rapid and specific for Cu^+. H. SHER

1784. **Study of indirect application of radioactive nuclei in analytical chemistry.** B. C. Purkayastha and V. R. P. Verneker (Inst. of Nucl. Phys., Calcutta). *J. Indian Chem. Soc.*, 1957, **34** (7), 487–493.—The determination of Ag and I by the use of ^{131}I is described. When zirconium phosphate is used as carrier, 1 μg of Ag can be determined to within ±2% in 30 min. The determination of Al in the presence of KI and KIO_3, the iodine liberated by hydrolysis being labelled with ^{131}I, is discussed. E. G. CUMMINS

1785. **Electro-migration on paper in the separation of ions. VII. [Separation of gold.]** H. G. Mukerjee (Scottish Church Coll., Calcutta). *Z. anal. Chem.*, 1957, **157** (6), 411–414 (in English).—Gold is successfully separated by paper electrophoresis from binary mixtures with certain other metals, namely Pt, Pd, Ru, Hg, Cu, Bi, Cd, Fe, Zn, Co and Ni. Suitable electrolytes are listed. With KSCN as electrolyte, Au is separated from Pt, Pd and Ru. The separations of Au from Pt and of Au from Ru are also achieved in oxalic acid and KCl, respectively. J. H. WATON

1786. **Determination of small quantities of beryllium with certain hydroxyquinones.** E. S. Przheval'skiĭ, T. A. Belyavskaya and A. P. Golovina. *Vestn. Moskov. Univ.*, 1956, (1), 191–196; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,833.—The possibility is indicated of determining Be photometrically by means of quinizarin (**I**), 5:8-dichloroquinizarin (**II**), naphthazarin (**III**), 1-amino-4-hydroxyanthraquinone (**IV**) and 1:4:5:8-tetrahydroxyanthraquinone (**V**). With **I** and **II** at pH ≃8, and with **III** at pH 5·57, Be forms complexes with max. absorption at 533 mμ. By use of compound **I** 10 to 60 μg, by compound **II** 1 to 100 μg, and by compound **III** 5 to 40 μg of Be per ml can be determined. Magnesium does not interfere when using **I** and **II**, but interferes when using **III**. The complex formed by Be with **IV** (at pH ≃8) has max. absorption at 574 mμ and can be used to determine 5 to 10 μg of Be per ml. Compound **V** forms with Be at pH 5·57 a complex which may be used to determine 0·1 to 10 μg of Be per ml. Magnesium does not interfere. Aluminium interferes with the determination in all cases but, when the ratio of Be to Al is 1:100, the influence of the Al may be removed by adding EDTA (disodium salt). A more sensitive method for the visual determination of 0·1 to 100 μg of Be depends on the fluorescence of the complexes in u.v. light. The determination is carried out by using the standard series method at pH ≃8 with **I**, **II** and **IV**, and at pH 5·57 with **III** and **V**. C. D. KOPKIN

1787. **Radiometric titration of the rare elements. Determination of beryllium, zirconium and thallium.** I. P. Alimarin, I. M. Gibalo and I. A. Sirotina (V. I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR). *Intern. J. Appl. Radiation Isotopes*, 1957, **2** (2), 117–128.—Beryllium (0·6 to 24 mg) was determined in a soln. buffered with acetate to between pH 5 and 5·5 by titration with 0·0913 *M* $(NH_4)_2HPO_4$ labelled with ^{32}P. The end-point was marked by the appearance of activity in the supernatant liquor. The ratio of Be to phosphate in the ppt. was 1·5. EDTA was used to mask Cu (381 mg), Fe (100 mg), Ca (40 mg), Mg (40 mg), Co (50 mg) and Ni (50 mg); Al (120 mg) was masked only if another of these elements was present. The error in 24 determinations was ≯4%. Beryllium was determined in bronze after dissolution in 50% HNO_3 and evaporation almost to dryness; and in beryllium concentrate after fusion with NaF and evaporation almost to dryness with H_2SO_4 (*cf. Anal. Abstr.*, 1957, **4**, 3575). Zirconium (2·7 to 29 mg) was similarly determined in 2 *N* acid by titration with 0·5 *M* H_3PO_4 labelled with ^{32}P. The ratio of Zr to phosphate in the ppt. was 1·0. Iron (1 g), Al (100 mg), Co (30 mg), Ni (500 mg) and Cu (40 mg) did not interfere; Ti did not interfere when H_2O_2 was added to the soln. The determination of Zr in iron-base alloys is described. Thallium labelled with ^{204}Tl was titrated with four reagents. With 0·05 *M* K_2CrO_4 the error in seven determinations (0·46 to 7·76 mg of Tl) was ≯3·2%. In the presence of EDTA at pH 4 a ratio of Pb to Tl of 50 can be tolerated. With 0·1 *M* tungstophosphoric acid, labelled with ^{32}P, a ppt. of $Tl_4H_3P(W_2O_7)_6$ was formed in 0·5 to 1 *M* HNO_3. Lead (124 mg), Hg (500 mg), Ag (17 mg) and Bi (10 mg) did not interfere with the determination of <2 mg of Tl in 1 *M* HNO_3. With 0·05 *M* sodium

tetraphenylboron the error in six determinations was $\pm 4{\cdot}3\%$. Thallium (0·4 mg) was titrated accurately in the presence of Pb (60 mg), in the presence of EDTA. Formulae are developed that enable the calculation of the end-point to be made from two measurements of the activity, instead of using the longer graphical method.

G. J. HUNTER

1788. **Separation of metal 8-hydroxyquinoline compounds between water and organic solvents. III. Photometric determination of magnesium by extraction of its oxinate with chloroform in the presence of amines.** F. Umland and W. Hoffmann (Inst. für Anorg. Chem., Tech. Hochschule, Hannover, Germany). *Anal. Chim. Acta*, 1957, **17** (2), 234–246 (in German).—The presence of primary or secondary *n*-butylamine (or their close homologues) permits the extraction of Mg as its oxinate with $CHCl_3$. The aq. phase should contain 0·05 to 10 μg of Mg and be buffered at pH 11·2 to 11·3. The Mg is then determined by measuring the extinction of the organic layer at 380 mμ. Interference at the working pH is shown by F^-, sulphosalicylic acid and EDTA. Interfering cations are either complexed with KCN or are removed by a pre-extraction without the addition of the butylamine. The method has been applied to the estimation of Mg in calcium minerals, and in aluminium and zinc alloys. Traces of Fe in the same material can be determined in the presence of many other metals by measuring the extinction at 580 mμ of the organic layer obtained during the pre-extraction with oxine and $CHCl_3$ alone.

J. H. WATON

1789. **Arsenate method for the iodimetric determination of calcium.** G. B. Shakhtakhtinskiĭ and G. A. Aslanov. *Azerb. Senae Inst. Eserleri, Trudy Azerb. Ind. Inst.*, 1956, (15), 103–110; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,801.—In the pptn. of Ca^{2+} from soln. of pure salts by arsenate ions, the influence is studied of excess of aq. NH_3, washing with aq. NH_3 of various concn., adding NH_4Cl and ammonium acetate, the time of standing after pptn., excess of precipitant and other factors. A new iodimetric method of determining Ca has been developed. To the weakly acid soln. add *N* Na_2HAsO_4 (20 ml) and NH_4Cl (1 to 2 g), dilute to about 100 ml and precipitate by stirring in conc. aq. NH_3. Add excess of the conc. aq. NH_3 (about 30 ml), filter off the ppt. after about 5 min., wash it 8 or 10 times with 4% aq. NH_3, transfer the filter to a conical flask, add H_2SO_4 (1:2·5) (about 25 ml), shake, add benzene (20 to 25 ml) and *N* KI (3 ml), shake vigorously for 20 to 30 sec., dilute with water (25 ml) and titrate with $Na_2S_2O_3$ soln. until the benzene layer is colourless. The arsenate method gives accurate results in a wide range of concn. of Ca and is more rapid than the permanganate method.

C. D. KOPKIN

1790. **Syntheses and analytical studies of colour reagents. I. Behaviour of oo′-dihydroxyazo compounds towards calcium and magnesium.** Koichi Emi, Kyoji Tôei and Haruo Miyata (Chem. Dept., Fac. Sci., Okayama Univ., Tsushima). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (6), 736–741.—One hundred *oo′*-dihydroxyazo compounds were synthesised by coupling 13 diazo components and 9 coupling components, and their colour reactions with Ca and Mg were examined. These dyes change their colour at about pH 10 and are used for detection at this value. Derivatives of 1-(*o*-hydroxyphenylazo)-2-naphthol and 1′;2-dihydroxy-1;2-azonaphthalene appear to have smaller limits of detection. 2-Amino-4-chlorophenol (**I**), 2-amino-4-chlorophenol-6-sulphonic acid and 1-amino-5-nitro-2-naphthol-4-sulphonic acid are useful diazo components, and chromotropic acid (**II**) seems to be the best coupling component. For the combination of **I** and **II**, the limit of detection is 0·002 μg of Ca and 0·04 μg of Mg per ml.

II. Behaviour of o-carboxy-o′-hydroxyazo compounds towards calcium. Koichi Emi, Kyoji Tôei and Nagaaki Tekemoto. *Ibid.*, 1957, **78** (6), 741–746.—*o*-Carboxy-*o′*-hydroxyazo compounds react with Ca in a basic soln. and produce a colour change. Magnesium is pptd. as hydroxide under the given conditions. The limit of detection was examined with 40 compounds. The reactivity depends mainly on the coupling component and little on the diazo component. This fact suggests that the linkage between Ca and the hydroxyl group of the coupling component is responsible for the colour change. The greater the acidity of this hydroxyl group, the stronger the linkage with Ca. R-acid appears to be the best coupling component; thus, 1-(2-carboxy-4-chlorophenylazo)-2-naphthol-3;6-disulphonic acid shows a distinctive change in colour from pink to orange in $<0{\cdot}4$ *N* NaOH in the presence of 1 μg of Ca per ml.

III. Behaviour of phthalein complexone reagents towards alkaline-earth ions. Koichi Emi, Kyoji Tôei and Tsuguyo Wada. *Ibid.*, 1957, **78** (7), 974–976.—Various phthalein complexone reagents were synthesised from iminodiacetic acid, formaldehyde and phthalein dyes such as cresolphthalein, *o*-chlorophenolphthalein, fluorescein, thymolphthalein or cresol red, and their reaction with Ba, Sr, Ca and Mg was studied. Those derived from dyes having a methyl, *iso*propyl or chloro group at the position ortho to the phenolic hydroxyl group give a colour change in the presence of alkaline-earth-metal ions in an ammoniacal soln. of pH $\simeq 10$.

IV. Behaviour of o-hydroxy-o′-sulphoazo compounds towards magnesium, calcium and barium. Koichi Emi, Kyoji Tôei and Haruo Miyata. *Ibid.*, 1957, **78** (7), 977–978.—Sixty *o*-hydroxy-*o′*-sulphoazo compounds were synthesised and their reactions with Mg, Ca and Ba in a dil. NaOH soln. of pH 10·5 (for Mg) and 12 (for Ca and Ba) were studied. The reagents change colour at pH 11 to 13. In the presence of alkaline-earth metals, they show acid colour at $pH < 11$. Those prepared with chromotropic acid (**I**) as the coupling component change from red to violet in the presence of these ions, whilst the others change from red to orange or *vice versa*. The sensitivity depends mainly on the coupling component, **I** and R-acid showing low limits of detection (**I**, $<0{\cdot}2$ μg per ml for Mg, $\simeq 1$ for Ca, 0·5 to 30 for Ba; R-acid, 1 to 5 for Mg, 0·5 to 2 for Ca, 3 to 30 for Ba).

V. Behaviour of o-trifluoromethyl-o′-hydroxyazo compounds towards alkaline-earth metals, thorium, aluminium, zirconium and uranium. Koichi Emi, Kyoji Tôei and Haruo Miyata. *Ibid.*, 1957, **78** (7), 979–980.—*o*-Trifluoromethylphenyldiazonium and *o*-tolyldiazonium salts were coupled with various naphthol derivatives and the reactions of the products with Mg, Ca, Ba, Zr, Th, Al and UO_2^{2+} were examined. The products are less sensitive towards alkaline-earth metals than are the *oo′*-dihydroxy- or *o*-hydroxy-*o′*-sulphoazo compounds. 2-(*o*-Trifluoromethylphenylazo)-1 : 8-dihydroxynaphthalene-3:6-disulphonic acid, amongst others, reacts with Al (limit of detection, 10 μg per ml, at pH 5 to 6), Zr (5 μg, at pH 1 to 2), Th (5 μg, at pH 3 to 4) and UO_2^{2+} (50 μg at pH 2 to 3).

K. SAITO

1791. **Micro-heterometric determination of cadmium with sodium diethyldithiocarbamate.** M. Bobtelsky and R. Rafailoff (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1957, **17** (2), 267–270 (in English).—In the heterometric titration described, 0·5 mg of Cd in 20 ml can be determined in 10 to 15 min. with an error of $\simeq 1\%$. The reaction is carried out in acetate soln., or preferably in ammoniacal citrate soln. In the presence of a suitable complexing reagent, a 100 to 1000-fold excess of Mg, Ca, Ba, Zn, Mn, Al, Cr^{III}, Fe^{III}, Sb^{III} or Pb does not interfere, nor is the sensitivity of the reaction diminished. J. H. WATON

1792. **New rapid gravimetric method for the determination of mercury.** E. Popper, N. Ariton, L. Proinov and R. Crăciuneanu. *Rev. Chim., Bucharest*, 1957, **8** (9), 594–596.—Mercuric ions are pptd. as a complex with an ethanolic soln. of 5-anilino-2-mercapto-1:3:4-thiadiazole. The method is accurate and sensitive down to concn. of 40 μg per ml, and is said to be suitable for application to pharmaceuticals. H. SHER

1793. **Separation and determination of traces of boron in silicon. I. Application of the chloroform extraction of tetraphenylarsonium fluoroborate.** L. Ducret and P. Seguin (Centre Nat. d'Études des Télécommun., Dépt. "P.C.M.", Issy-les-Moulineaux, France). *Anal. Chim. Acta*, 1957, **17** (2), 207–212 (in French).—The extraction of B as tetraphenylarsonium fluoroborate (*cf.* Coursier *et al.*, *Anal. Abstr.*, 1956, **3**, 661), followed by the colorimetric determination with curcumin (*cf.* Philipson, *LantbrHögsk. Ann.*, 1944–5, **12**, 251), is applied to the determination of B in silicon.

II. New method for separation and determination of boron. Application to the determination of boron in silicon. L. Ducret. *Ibid.*, 1957, **17** (2), 213–219 (in French).—Boron is extracted as the fluoroborate of methylene blue by 1:2-dichloroethane, and the extinction of the organic layer is measured at 645 mμ. The procedure has been applied to the estimation of B in water, silicon and silica. J. H. WATON

1794. **Chromoxan pure blue B as a reagent for aluminium.** F. R. Sheyanova and V. P. Malenskaya (N. I. Lobachevskiĭ Gorki State Univ.). *Zavod. Lab.*, 1957, **23** (8), 907–909.—Chromoxan pure blue B gives a violet colour with salts of Al in neutral or weakly acid soln. In more strongly acid soln. (pH 2) the colour appears on heating. The determination of Al can be carried out photometrically at 540 to 550 mμ; the optimum pH is 6, and Mg, Zn and Mn do not interfere. The method is sensitive to 5 μg of Al in 1 ml and can be used for determining up to 8% of Al in magnesium and zinc alloys. G. S. SMITH

1795. **Spectrophotometric determination of micro amounts of aluminium and iron in alkalis with 8-hydroxyquinoline.** Hiroshi Awaya, Senpachi Miyoshi and Kenji Motojima (Tokuyama Soda Co., Yamaguchi Prefecture). *Japan Analyst*, 1957, **6** (8), 503–507.—Motojima's simultaneous photometric determination of Al and Fe by extraction with 8-hydroxyquinoline (**I**) into $CHCl_3$ (*Anal. Abstr.*, 1956, **3**, 1725) was used in a modification of Kenyon's method (*Brit. Abstr. C*, 1953, 108) for the determination of Al and Fe in caustic alkali. The presence of <4 g of NaCl does not vitiate the determination of <28 μg of Al and <100 μg of Fe. Sample soln. and aq. NH_3 soln. must be stored in polyethylene bottles. The results for Fe agree well with those obtained by the separation of Fe as hydroxide followed by colorimetry with SCN^-, whereas those for Al are markedly lower than those calculated from the difference between the weight of ignited mixed oxides of Al and Fe and the amount of Fe determined colorimetrically. By a careful examination of these methods, it was found that the results obtained by the present method are correct. The time taken for an estimation is $\simeq 30$ min. *Procedure*—Neutralise the sample with 6 *N* HCl, acidify to give $\simeq 0{\cdot}1$ *N* HCl soln. and make up to 100 or 250 ml. Filter off any insol. matter. To an aliquot (<35 ml) containing 10 to 150 μg of Fe and 5 to 50 μg of Al add **I** soln. (0·1% in acetic acid) (3 ml) and 2 *N* ammonium acetate (3 ml) and adjust the pH to between 5·0 and 5·5 with 2 *N* aq. NH_3; dilute to 50 ml and shake with $CHCl_3$ (10 ml) for 1 min. Dry the $CHCl_3$ layer with Na_2SO_4 (1 g) and measure the extinction at 390 and 470 mμ. Calculate the amount of Al and Fe by the empirical formula given previously (*loc. cit.*). K. SAITO

1796. **Determination of aluminium and copper in die-cast zinc alloys by the use of anion-exchange resin.** Masuo Kojima (Chem. Dept., College of Gen. Educ., Tokyo Univ., Meguro-ku). *Japan Analyst*, 1957, **6** (6), 369–375.—Aluminium ($\simeq 4\%$) in zinc is co-pptd. with a small amount of Zn at pH 5, the ppt. is dissolved in 2 *N* HCl (30 ml) and the soln. is passed through a column (diam., 9 mm; length, 11 cm) of Dowex-2 (Cl) resin (60 to 80 mesh) at 2 ml per min. Zinc is quant. adsorbed as the chloro complex, and Al in the eluate is treated with aq. NH_3 and 0·05 *M* EDTA and titrated with $Th(NO_3)_4$, with alizarin red S as indicator. If the alloy contains $\simeq 1\%$ of Cu, the sample soln. is made 1·77 *N* in HCl containing 65% ethanol and passed through the resin column; Zn and Cu are adsorbed and Al is determined in the eluate as described above. The K_d value of Cu increases markedly with ethanol content. Copper is eluted from the column, together with a small amount of Fe, with 2 *N* HCl, and is titrated with EDTA in an ammoniacal soln., with murexide as indicator; Fe is pptd. as $Fe(OH)_3$. Small amounts of Cd, Sn and Pb in the alloy do not interfere with the separation. The results agree well with those by the conventional methods. K. SAITO

1797. **Spark-spectrographic determination of light alloy metals with spectrophotometer. I. Analysis of aluminium alloys by high-voltage spark.** Motoyaka Mutaguchi and Kazuo Tasuda (Casting Res. Lab., Waseda Univ., Shinjuku-ku, Tokyo). *Japan Analyst*, 1957, **6** (6), 349–355.—The stability of various light sources for use in emission spectroscopic analysis with the Beckman DU spectrophotometer was examined. A condensed spark (10,500 V, 0·033 μF) between a thin rod of sample (diam. 1 mm at the top, lower electrode) and a pure carbon rod (diam. 4 to 5 mm) provides a stable source for the determination of Cu (3247 A, 0·5 to 3%), Mg (2795 A, 0·15 to 3%), Mn (2593 A, 0·2 to 1%) and Si (2881 A, 3 to 12%) in aluminium alloys. The intensity of each line changes with time but is stable for several minutes after a preliminary discharge for a certain period, namely, 5 min. for Mn and Si, 10 min. for Cu, and 15 min. for Mg. The use of a concave mirror is recommended for condensing the light. K. SAITO

1798. **Systematic analysis of aluminium alloys, using chromatography, by elution from cellulose columns. I. Qualitative analysis.** G. Venturello and A. M. Ghe (Univ. Bologna). *Ann. Chim., Roma*, 1957, **47** (7–8), 912–918.—A full description of the method is given, together with tables of results achieved with different solvents. Successive elutions were used to separate the elements retained in the column (Al, Mn, Ni, Mg and Cr). The first eluate is then passed through a second column, which is subsequently eluted to separate the remaining elements (Cu, Zn and Fe). It has proved possible to detect the most important elements—especially Mn, Mg, Cu, Fe, Zn, Ni and Cr—in aluminium alloys in amounts down to 0·1%.

II. Quantitative analysis. G. Venturello and A. M. Ghe. *Ibid.*, 1957, **47** (7–8), 919–928.—By using the same methods as were described in Part I, quantitative analyses can be carried out. The determination is claimed to be suitable for concn. of Ni, Zn and Cr down to 0·1%, and of Mn and Mg down to 3%, but for Fe and Cu the limits are higher. Many of the difficulties caused by interfering ions in other methods of analysis are avoided. The results of several experiments are given, and the accuracy for any given element is claimed to be of the order of ±0·02 to ±0·04% of the total amount of that element present in the alloy.

J. F. P. H. GREENE

1799. **Thermal decomposition of scandium, yttrium and rare-earth-metal oxalates.** W. W. Wendlandt (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 58–61.—The thermal decomposition of the oxalates of Sc, Y, La, Ce^{3+}, Pr, Nd, Sm, Eu, Gd, Ho and Er is described and discussed, with reference to their composition and that of their hydrates. E. G. CUMMINS

1800. **Determination of carbon in titanium and zirconium.** W. T. Elwell and D. F. Wood (I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Analyst*, 1957, **82**, 769.—In an extensive examination of the determination of C in titanium with the use of fluxes and recommended conditions of oxidation it was found that oxidation of the prepared samples can be easily controlled if the combustion tube is first filled with argon. The determination then follows the conventional method, the CO_2 being determined gravimetrically or conductimetrically. In the gravimetric method a large sample wt. may be used. The method has been successfully used for titanium and its alloys in the presence of a lead flux. With zirconium and its alloys and the proposed modification a flux need not be used. A. O. JONES

1801. **The preparation and analysis of carbon-14-labelled cyanide.** J. D. Moyer and H. S. Isbell (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. NBS-4708, 1956, 20 pp.—Detailed directions, based on the method of Sixma *et al.* (*Rec. Trav. Chim. Pays-Bas*, 1954, **73**, 161), are given for the preparation of $Na^{14}CN$ from $Ba^{14}CO_3$ in 90 to 95% yield. Simple reliable methods are presented for the analysis of ^{14}C-labelled alkali cyanide and $BaCO_3$. The labelled cyanide is analysed for (*a*) total radioactivity, (*b*) radioactivity in the form of cyanide, and (*c*) specific radioactivity. The procedures are based on reaction of the cyanide with a reducing sugar, and measurement of fixed C in either formamide or alkaline ethanediol soln. by means of a proportional counter. In the analysis of $Ba^{14}CO_3$ and other materials, CO_2 formed either by acid treatment or wet oxidation is absorbed in alkaline ethanediol. When carbonates are decomposed by acid, the last traces of labelled CO_2 are swept away from the apparatus by the addition and slow decomposition of ethylene carbonate; the ethanediol soln. is then counted directly in a proportional counter.

CHEM. ABSTR.

1802. **Coulometric titrations with Hg^+ and Hg^{2+}. Determination of cyanide.** E. P. Przybylowicz and L. B. Rogers (M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 65–69.—The coulometric generation of Hg^{2+} was successfully carried out in dil. alkaline cyanide buffered at pH 9·2. Both direct titration and pre-titration methods are described and the former is rejected. Cyanide at the 1×10^{-5} *N* level is detectable with an error of 1·9% when Hg^{2+} are used, whereas reported results with Ag, at the 4×10^{-5} *N* cyanide level, show an error of 2·3%. E. G. CUMMINS

1803. **Spectrographic determination of germanium in coal ash.** N. V. Arnautov. *Izv. Vost. Fil. Akad. Nauk, SSSR*, 1957, (1), 49–52; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,842.—Join a copper tube of diam. 3 mm and length 5 mm to a copper rod sharpened to a truncated cone. In the annular space so formed place a mixture of coal ash and powdered spectrally pure carbon in the ratio 1:2 (25 to 30 mg). Introduce 2% of Cd into the sample as CdI_2. As upper electrode use a 3·5-mm-diam. copper rod sharpened to a cone. Excite the spectrum in an a.c. arc discharge at 5 amp. and arc gap 2·5 mm. Photograph the spectra on a medium spectrograph with a 0·02-mm slit. The exposure is 10 to 20 sec. according to the Ge content. The analytical lines used are Ge 3039·06 and Cd 2980·63 A. As comparison element it is possible to use the background, with an insignificant increase in error. Construct the calibration curves on the co-ordinates ΔS and log *C*. The sensitivity of the analysis is 0·0003%. Prepare standards from Ge-free ash with GeO_2 added. The error is ±5% with triple photography of the spectra. C. D. KOPKIN

1804. **Volumetric determination of tin in titanium and its alloys.** H. J. G. Challis and J. T. Jones (I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Analyst*, 1957, **82**, 658–663.—The sample (0·2 g if the content of Sn is >0·5%; 0·5 g if the Sn is 0·2 to 0·5%) is dissolved in dil. H_2SO_4, the cooled soln. is oxidised by the dropwise addition of HNO_3 and heated until the Sn is dissolved. The soln. is evaporated to fuming-point, then treated with water, conc. HCl, 1 ml of satd. $HgCl_2$ soln. and 5 g of NaH_2PO_2. The liquid is boiled in a flask with a delivery tube leading into a 40% soln. of $NaHCO_3$ and the flask is cooled with the tube under the surface of the soln. The cold liquid is treated with 2 g of KI, the stoppered flask is shaken and the contents are titrated with 0·02 *N* KIO_3, starch being used as indicator. A modification for finely divided drillings that avoids the use of H_2SO_4 is described. In a modification for amounts of Sn < 0·2% the sample is dissolved in dil. H_2SO_4, oxidised with HNO_3 under specified conditions, $CdSO_4$ is added as a carrier, the Sn is pptd. by H_2S, the collected ppt. is dissolved in HCl, $Ti(SO_4)_2$, $HgCl_2$ and NaH_2PO_2 are added, the Sn is reduced as before and titrated with 0·005 *N* KIO_3. Recovery of 0·002% of Sn is satisfactory. No interference occurs from likely alloying metals.

A. O. JONES

1805. **Spectrochemical analysis of white metals by the point-to-plane spark technique.** H. K. Nausester (Spectrochemical Lab., The Canada Metal Co. Ltd., Toronto, Ontario). *Appl. Spectroscopy*, 1957, **11** (3), 115–119.—A controlled spark source, with series gap, and a 1·5-metre grating spectrograph are used for the analysis of a wide range of tin- and lead-base alloys. Full details of exposure conditions and line pairs are given. Both major constituents, such as Sn up to 20% and Sb up to 28% in type metals and Sn up to 60% in solders, and the lower constituents and impurities can be determined. Matrix and background corrections are used, and comparative spectrographic and chemical analyses of typical alloys are given to show the accuracy to be expected. The importance of the method of taking the chill-cast disc sample is emphasised. A typical antimonial lead disc sample, containing 6·82% of Sb, 0·36% of Sn, 0·00043% of Cu, 0·40% of As and 0·0048% of Ag, was sparked at 8 positions throughout its usable volume, and gave coeff. of variation of 2·6, 5·2, 3·3, 5·0 and 4·2, respectively. P. T. BEALE

1806. **Complexometric determination of lead in lead concentrates.** L. B. Zaĭchikova, N. N. Lutenko and V. P. Ioffe (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1957, **23** (8), 910–911.—With low contents of Ba (<2%), the material is treated with HCl and HNO_3 followed by fuming with H_2SO_4, the $PbSO_4$ is collected and dissolved in ammonium or Na acetate, and the Pb is titrated with EDTA (disodium salt), with Eriochrome black T as indicator. With large amounts of Ba (>2%), the material is treated with HCl and NaCl and the soln. is filtered before addition of H_2SO_4.
G. S. SMITH

1807. **Colour reactions of titanium with phenols.** S. Ya. Shnaĭderman. *Ukr. Khim. Zhur.*, 1957, **23** (1), 92–96; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,802.—The colour reactions of Ti^{4+} with chromotropic acid (**I**), catechol (**II**), pyrogallol (**III**) and gallic acid (**IV**) were studied. At pH 3 to 4, Ti^{4+} forms with **I** a complex with the mol. ratio Ti:**I** of 1:2. Within 2 to 5 days this ratio becomes 1:4. At pH 8, a complex with ratio Ti:**I** of 1:2 is formed, this ratio remaining unchanged during 5 days. With **II**, **III** and **IV**, Ti^{4+} in a weakly acid medium forms complexes with ratios of Ti:reagent equal to 1:4, 1:4 and 1:2, respectively. In conc. soln. of H_2SO_4, Ti^{4+} forms with **I**, **II**, **III** and **IV** complexes of a clear red colour. The optical density of soln. of the complexes with **II**, **III** and **IV** (blue filter, $\lambda_{max.}$ 420 mμ) and with **I** (green filter, $\lambda_{max.}$ 530 mμ) has a max. value in 90% H_2SO_4 and remains const. in the presence of <15-fold excess of **II**, <25-fold excess of **III** and **IV**, and <10-fold excess of **I**. In conc. soln. of H_2SO_4, Ti^{4+} forms complexes with **I**, **II**, **III** and **IV** in the ratios 1:1, 1:4, 1:2 and 1:4, respectively. The intensity of the colour of the complexes in H_2SO_4 soln. is greater than in HCl soln.
C. D. KOPKIN

1808. **Spectrophotometric determination of titanium with phenylfluorone.** V. Damodaran (Nat. Chem. Lab., Poona). *J. Sci. Ind. Res., B, India*, 1957, **16** (8), 366–369.—The micro-determination described is based on the formation of a pink complex of Ti^{4+} with phenylfluorone in the pH range 1·9 to 3·1. The method is applicable to soln. too dilute for the use of H_2O_2 or tiron. Uranium and Th do not interfere, but oxalate, phosphate and Zr interfere to various extents. The interference of Fe^{3+} and V can be eliminated with ascorbic acid, and molybdate or tungstate should be removed by the pptn. of Ti in the presence of Fe as a collector with excess of KOH, and the ppt. fused with pyrosulphate. In general, only elements that form ppt. or colloidal soln. of the respective hydrous oxides at pH <3 interfere by giving coloured complexes with the reagent. I. JONES

1809. **Fast grey RA, a new colorimetric reagent. I. Spectrophotometric micro-determination of zirconium.** H. Khalifa and M. R. Zaki (II Chem. Inst., Univ. Wien). *Z. anal. Chem.*, 1957, **158** (1), 1–7.—Fast grey RA (**I**) forms a purple complex with Zr which can be used for the determination of small amounts (0·2 to 80 μg) of Zr. Rare-earth and alkali metals, U, Co, Al, Th, Bi and Zn do not interfere. Ascorbic acid can be used to mask the interference of small amounts of Fe. Attempts to mask Cu, Cd, Ni and large amounts of Fe were not successful. *Procedure*—Prepare a soln. of the sample in 0·1 *N* HCl (1 ml). If the amount of Zr is <10 μg, add 0·4 *N* HCl (5 ml) and a freshly prepared 0·005% aq. soln. of **I** (3 ml); if the amount of Zr is >10 μg, add 0·1 *N* HCl (4 ml) and 0·05% **I** (3 ml). Dilute with water to 10 ml, mix, and after 20 to 50 min. measure the extinction at 570 mμ against a reagent blank. A. R. ROGERS

1810. **Analytical aspects of some organic compounds. VI. 2-Hydroxy-3-naphthoic acids in the determination of thorium and zirconium.** S. K. Datta (Darjeeling Gov. Coll., India). *J. Indian Chem. Soc.*, 1957, **34** (7), 531–536.—2-Hydroxy-3-naphthoic acid and several of its deriv. have been used for the determination of Zr and Th. Thorium may be separated from the cerium earths, and Th and Zr from foreign ions, by means of the bromo, iodo, nitro and nitroso deriv., which may also be used for the extraction of Th from monazite sands. The bromo, iodo and nitro deriv. may also be used for the separation of Th from U. *Procedure*—A soln. of Th or Zr is made neutral to Congo red and then heated to 80°. To this soln. is added, with stirring, a 1% soln. of reagent followed by 2% ammonium acetate soln. (1 ml). The reagents used are 2-hydroxy- (**I**), 1-bromo-2-hydroxy- (**II**), 2-hydroxy-1:6-di-iodo- (**III**), 2-acetyl- (**IV**), 2-hydroxy-4:6-dinitro- (**V**), and 2-hydroxy-1-nitroso-naphthoic acids (**VI**). For **V** and **VI**, a 1% soln. in hot water was used, but for the other deriv. soln. in 75% ethanol were used. The mixture is heated for 5 min. on a boiling-water bath to coagulate the ppt. **I**, **II** and **III** give yellow ppt., **IV** white, and **V** and **VI** deep yellow. The ppt. is filtered off, washed with hot water and then ethanol, dried at 110° and ignited to ThO_2 or ZrO_2. The accuracy of the method varies slightly with the reagents but is always within ±3% for amounts of ThO_2 or ZrO_2 >15·0 mg, and is considerably higher for 5 mg.
C. A. SLATER

1811. **Determination of small quantities of nitric oxide in gases.** L. M. Kontorovich. *Trudy Gos. Nauch. i Proektn. Inst. Azotn. Prom.*, 1954, (3), 218–224; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,818.—The permanganate method of determining small quantities of NO in gases has been tested on gaseous mixtures containing 1% of O and 1 to 10 ml of NO per 10 litres of gas. Pass the gas mixture through an absorber with the Griess reagent to remove NO_2, and then at a rate of 20 to 25 litres per hour through an oxidising soln. comprising 50 ml of a 5% soln. of $KMnO_4$ and 5 ml of H_2SO_4 (1:1), to oxidise NO to NO_2, which is then trapped in two absorbers placed

in series containing 15 ml each of the Griess reagent. Because of the high intensity of colour formed, the gas is passed through the two absorbers in turn and the solutions are subjected to colorimetry, the results being added. The standard scale for the colorimetry is prepared from the Griess reagent with additions of various quantities of standard soln. of $NaNO_2$. The analysis takes 0·5 to 2 hr.; the error is ±13%. The method may be used for determining NO in coke gas; in this case the $KMnO_4$ soln. in the absorbers is changed after each determination. If the gas contains large quantities of reducing substances (olefins and diolefins), oxidising agents of the flowing type are used instead of absorbers.

C. D. KOPKIN

1812. **Spectrophotometric determination of nitrite and thiourea.** K. Hutchinson and D. F. Boltz (Wayne State Univ., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 54–56.—The absorbance at 455 mμ of the Fe^{3+} - thiocyanate complex $FeSCN^{2+}$ was used to determine the thiocyanic acid formed by reaction between nitrite and thiourea. The ranges 2 to 12 p.p.m. of nitrite or 4 to 32 p.p.m. of thiourea have been successfully covered in the presence of excess of thiourea or nitrite, respectively. The interfering ions listed range from Ni^{2+} with a tolerance of 150 p.p.m., through Cr^{2+}, VO_2^-, Cd^{2+}, F^-, PO_4^{3-}, $C_2O_4^{2-}$, CO_3^{2-}, AsO_4^{3-} to I^-, 5 p.p.m. The method is less sensitive than the classical Griess method.

E. G. CUMMINS

1813. **Determination of small amounts of pyrophosphate in soluble orthophosphates.** W. B. Chess and D. N. Bernhart (Victor Chemical Works, Chicago Heights, Ill., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 111–112.—The method is based on the replacement of the pyrophosphate in the Fe^{3+} - pyrophosphate complex with 1:10-phenanthroline, and the subsequent reduction by hydroxylamine to the coloured ferrous complex. The reaction should be carried out at 30° to 40°, and the extinction is measured at 515 mμ. The results were accurate to within ±0·04%.

E. G. CUMMINS

1814. ***o*-Dithiols in analysis. V. The zinc complex of toluene-3:4-dithiol as a reagent for arsenic and germanium.** R. E. D. Clark (Dept. of Sci. and Technology, Tech. Coll. and School of Art, Cambridge, England). *Analyst*, 1957, **82**, 760–763.—When the zinc complex of toluene-3:4-dithiol (zinc dithiol) is sprinkled into hot HCl and the soln. is boiled, the toluene-3:4-dithiol is liberated rapidly in the steam. Arsenic and germanium are the only cations that form colourless or nearly colourless complexes with toluene-3:4-dithiol in strongly acid soln., and, in the presence of strong reducing agents, the formation of a turbidity is selective for As^V (reduced to As^{III}) and Ge. The test soln. (1 ml) is treated with conc. HCl (0·5 ml) and 50 to 150 mg of $SnCl_2$, the liquid is boiled, zinc dithiol is sprinkled on the surface and boiling is resumed for 10 to 20 sec. A permanent turbidity indicates As. If Ge is present, the original test soln. is first treated with $KClO_3$ and evaporated twice to a third of its vol. with HCl. To test for Ge, the acidity of the test soln. is adjusted to between 2·5 and 3·5 *N* with HCl, $KClO_3$ is added, the liquid is boiled and 50 ml of the vapour is withdrawn by means of a teat pipette. The condensate is treated with half its vol. of HCl and 50 to 150 mg of $SnCl_2$. The liquid is boiled, zinc dithiol is sprinkled on the surface and the mixture is re-heated. A white turbidity indicates Ge. Tungstic acid and some of the less common elements interfere.

A. O. JONES

1815. **Titration of arsenate ions with decimolar magnesium sulphate solution in the presence of calcium, magnesium, ferric and cupric ions and some other foreign substances.** E. Bakács, L. Szekeres and B. Láng (Inst. of General and Inorg. Chem., Agric. Univ., Budapest). *Z. anal. Chem.*, 1957, **158** (1), 14–20 (in English).—The method described previously (*cf. Anal. Abstr.*, 1957, **4**, 1179) for the titration of AsO_4^{3-} has been modified to permit the convenient analysis of technical materials such as plant-protecting suspensions. The coeff. of variation based on 20 assays is 0·57%. *Procedure*—Extract the homogenised sample (5 g) with 4 *N* HCl (40 ml) and 2 *N* HCl (50 ml) in small portions, filter, and dilute the filtrate to 125 ml with H_2O. Add ascorbic acid (0·1 or 0·2 g) and 0·1% methyl red soln. (2 drops) to a 5-ml aliquot and after 10 min. neutralise with 10% aq. NH_3. Add 5% KCN soln. (1 ml), 0·1 *M* EDTA (disodium salt) (15 ml) and NH_4Cl - aq. NH_3 buffer of pH 10 (3 ml). After 10 min., add 0·25% Eriochrome black T soln. (10 drops) and immediately titrate with 0·1 *M* $MgSO_4$ to a purple - violet colour. Add 90% ethanol (15 ml) and titrate the free AsO_4^{3-} with 0·1 *M* $MgSO_4$ as described previously (*loc. cit.*).

A. R. ROGERS

1816. **Irreversible redox indicators. I. Study of the photometric method of determining antimony by titration with bromate in the presence of methyl orange or methyl red as irreversible redox indicator.** C. Liteanu and I. Máthé. *Rev. Chim., Bucharest*, 1957, **8** (8), 540–543.—The influence of the concn. of Br^-, H^+, HCl and light on the speed of decolorisation of the indicator has been studied, in order to eliminate titration errors caused by these factors. For a rapid sharp end-point, the concn. of HCl should lie between 6 and 10%, but at that concn. decolorisation of the indicator takes place before the end-point is reached. The addition of KBr delays decolorisation, but the concn. of Sb^{3+} must be so arranged that the concn. of Br^- resulting from the reaction is ≯0·031 moles per litre. Methyl orange is preferred to methyl red, because of its greater solubility in water, and more rapid decolorisation.

H. SHER

1817. **Photometric determination of antimony in pure and electrolytic copper.** C. M. Dozinel (1, Place Fleur de Blé, Brussels). *Z. anal. Chem.*, 1957, **157** (6), 401–405.—As little as 0·0001 to 0·003% of Sb in copper can be determined colorimetrically with rhodamine B after extraction with di*iso*propyl ether. The sample is dissolved in HCl and H_2O_2, and is treated with Na_2SO_3 and then $Ce(SO_4)_2$ to ensure that all the Sb is present as Sb^V. After extraction of Sb with di*iso*propyl ether, the organic layer is shaken with aq. rhodamine B soln. (0·04 g in 100 ml of *N* HCl), and then the extinction is measured relative to that obtained from an antimony-free copper or a copper of known antimony content. The procedure is rapid, requiring <30 min.

J. H. WATON

1818. **Determination of bismuth in lead and lead alloys.** N. W. Fletcher and R. Wardle (The Chloride Electrical Storage Co. Ltd., Exide Works, Swinton, Manchester, England). *Analyst*, 1957, **82**, 747–750.—The sample is dissolved with gentle heating in a mixture of Br and HBr, $HClO_4$ is added and the liquid is boiled until $PbBr_2$ begins to crystallise. Heating is continued with swirling (to prevent spilling and bumping) until the vol. is reduced to 2 or 3 ml. If necessary the treatment with HBr, Br and $HClO_4$ and the evaporation are repeated.

To the cooled soln. HBr is added and then ascorbic acid, and the soln. is diluted to a known vol. with water and the temp. is adjusted to 20° ± 2°. The extinction is measured at 375 mμ with a violet filter against water, and the extinction of a reagent blank is measured similarly. The content of Bi is then ascertained from a calibration graph. If Te is present, free Br in the original sample soln. is removed by boiling and the Te is pptd. with $SnBr_2$. The ppt. is removed by filtration, the filtrate is treated with $HClO_4$ and the procedure is then as already described. Results of the analysis of the same alloy by different analysts showed satisfactory reproducibility. The wt. of sample is limited to 1 g and the lower limit of the method is 0·001%.

A. O. JONES

1819. **Ion-exchange separations in the analysis of bismuth-base alloys. I. Binary alloys with uranium and thorium.** G. W. C. Milner and J. W. Edwards (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1957, **17** (2), 259–263 (in English).—Macro amounts of Bi are separated from small amounts of Th or U by adsorption on the anion-exchange resin De-Acidite FF. In this way, a preliminary separation of Bi can be effected in the determination of Th and U in binary mixtures with Bi. After the removal of organic matter by wet oxidation, milligram amounts of Th are determined volumetrically with EDTA (disodium salt) and catechol violet, and milligram amounts of U with $Ce(SO_4)_2$ after reduction on a lead column. Microgram amounts of each element are determined absorptiometrically with thoronol. J. H. WATON

1820. **Determination of vanadium in deposits on surfaces of boilers and turbine blades.** K. I. Sekt and T. P. Kuznetsova (All-Union Thermotech. Sci. Res. Inst.). *Zavod. Lab.*, 1957, **23** (8), 918.—The V is oxidised with $KMnO_4$ and titrated with 0·02 *N* Fe^{III} in the presence of *N*-phenylanthranilic acid. G. S. SMITH

1821. **Separation of niobium and tantalum with N-benzoyl-N-phenylhydroxylamine.** A. K. Majumdar and A. K. Mukherjee (Jadavpur Univ., Calcutta). *Naturwissenschaften*, 1957, **44** (18), 491 (in English).—Niobium is pptd. with the reagent as the oxide at a pH between 3·5 and 6·5. Tantalum is pptd. from the filtrate by the same reagent by adjusting the pH to a low value by the addition of dilute H_2SO_4. A time of 45 minutes is required for complete pptn. Other ions, except those of titanium, zirconium and vanadate, are kept in solution by a mixture of tartrate and EDTA.

E. KAWERAU

1822. **Separation of niobium and tantalum [by sublimation of their halides obtained by action of aluminium halides on their oxides].** M. Chaigneau. *Compt. Rend.*, 1957, **244** (7), 900–902.—Mixtures of Nb_2O_5 and Ta_2O_5 with one of the aluminium halides are heated in a closed tube at 200° to 400° for 24 to 48 hr. After removal of gaseous products, the residues are sublimed (pressure not stated). The niobium and tantalum halides are separable, owing to their different volatilities. Sublimation temp. are: $TaCl_5$ 150°, $NbOCl_3$ 230°; $NbBr_5$ 220°, $TaBr_5$ 300° (all under vacuum); TaI_5 540°, NbI_3 non-volatile (at atm. pressure). The most promising halide appears to be AlI_3. D. A. PANTONY

1823. **Analysis of peroxy compounds. I. Determination of the constitution of peroxy compounds.** L. J. Csányi and F. Solymosi (Inst. für Anorg. und Anal. Chem., Univ., Szeged). *Acta Chim. Acad. Sci. Hung.*, 1957, **13** (1–2), 9–17 (in German).—The following procedure is suggested for the analytical identification of the peroxide –O–O–, hydroperoxide –O–O–H and peroxy di-acid groupings. By shaking the sample with ether, H_2O_2 is separated from its addition compounds, and after extracting the H_2O_2 into water it can be identified by the addition of K H phthalate, which is oxidised in the presence of $FeSO_4$ to give a yellow - brown colour. The evolution of Br after the addition of KBr to the soln. indicates the presence of a hydroperoxide. In the identification of peroxy di-acids, H_2O_2 and hydroperoxides are removed by adding an excess of arsenite, which, catalysed by a few drops of osmium tetroxide, reacts with H_2O_2 to form arsenate. The unchanged arsenite is removed by the addition of $KMnO_4$ or $Ce(SO_4)_2$. The presence of peroxy di-acids is indicated by the evolution of Br on warming with KBr. The readiness with which these substances decompose must be borne in mind when interpreting results.

II. Induced reactions occurring in the analysis of H_2O_2 - H_2SO_4 systems. L. J. Csányi and F. Solymosi. *Ibid.*, 1957, **13** (1–2), 19–37.—A significant error occurs in the amount of H_2O_2 and peroxysulphuric acids found when determined by $KMnO_4$ or $Ce(SO_4)_2$. The ratio of the error of H_2O_2 and the peroxysulphuric acids is $\simeq 1$, which indicates that the titration reaction, which was found to be catalysed by Mn^{2+}, induces a mole per mole reaction between H_2O_2 and peroxysulphuric acids. This partly confirms Friend's findings (*J. Chem. Soc.*, 1904, **85**, 527; 1905, **87**, 1367; 1906, **89**, 1092) and invalidates those of Skrabal and Vacek (*Öst. Chem-Ztg*, 1901, **13**, 27). The errors can be minimised by increasing the acid concn., carrying out the titration more rapidly and at a low temp., reducing the vol. of soln., and by adding a large amount of Mn^{2+}.

III. Cerimetric determination of hydrogen peroxide, peroxymonosulphuric acid (Caro's acid) and peroxydisulphuric acid in the presence of each other. L. J. Csányi and F. Solymosi. *Ibid.*, 1958, **13** (3–4), 257–273.—The sample containing H_2O_2, H_2SO_5 and $H_2S_2O_8$ is made *N* with H_2SO_4 and treated with 0·1 *N* H_2AsO_3, and the H_2O_2 is titrated with $Ce(SO_4)_2$ soln., with ferroin as indicator. The H_2SO_5 is reduced by the H_2AsO_3 and thus the possibility of an induction error is excluded. After the addition of a drop of osmic acid as catalyst, the excess of H_2AsO_3 is titrated with $Ce(SO_4)_2$. The determination of $H_2S_2O_8$ depends on making the soln. 2 to 3 *N* with H_2SO_4 and boiling for 6 to 8 min. in an atmosphere of CO_2 in the presence of excess of 0·1 *N* H_2AsO_3 and titrating the excess with $Ce(SO_4)_2$ soln.

IV. Cerimetric determination of hydrogen peroxide and peroxyacetic acid and of hydrogen peroxide and peroxyphosphoric acid when present together. L. J. Csányi and F. Solymosi. *Ibid.*, 1958, **13**, (3–4), 275–282.—The method described in Part III has been applied to H_2O_2 - peroxyacetic acid and H_2O_2 - H_3PO_5 - $H_4P_2O_8$. The interference of PO_4^{3-} is avoided by the addition of $Al_2(SO_4)_3$. F. J. M.

J. S. HETMAN

1824. **Determination of oxygen in titanium and titanium alloys, based on the principle of chlorination.** W. T. Elwell and D. M. Peake (I.C.I. Ltd., Metals Div., Kynoch Works, Birmingham, England). *Analyst*, 1957, **82**, 734–742.—In the apparatus described, the solid sample is mixed with graphite and heated in a stream of Cl in an atmosphere of argon. The resulting mixture of CO and excess of Cl is passed through a tube containing antimony

sticks in which the Cl is absorbed. The residual CO is then oxidised by passage over heated CuO and the CO_2 is collected in a tared absorption tube containing Carbosorb soda asbestos and anhydrone. Results, compared with those obtained with the much more expensive vacuum fusion apparatus, indicate satisfactory accuracy. Good reproducibility was obtained when the method was applied to the analysis of Ti - Mn alloys to which the vacuum fusion method is not applicable. With modification, chiefly to prevent blockage of the Cl trap with $ZrCl_4$, the method is probably applicable to the determination of O in zirconium and its alloys.

A. O. JONES

1825. **Micro-determination of sulphur in iron and steel. I. Photometric determination by the potassium dichromate - diphenylcarbazide method.** Akira Sakihama (Res. Lab., Kobe Steel Works Ltd., Nada-ku). *Japan Analyst*, 1957, **6** (7), 439–442.—Vaughan and Whalley's colorimetric method (*J. Iron & Steel Inst.*, 1947, **155**, 535) was modified for micro-determinations (<20 mg of the sample, error <0·004% of S). The absorption of the SO_2 by $K_2Cr_2O_7$ soln. is incomplete, presumably owing to the influence of dissolved O, but the ratio of absorbed S to total S remains practically constant when a 0·4 *N* HCl soln. of $K_2Cr_2O_7$ is used after combustion at 1350° in a stream of O at 50 to 75 ml per min. *Procedure*—Insert the sample (≃10 mg) in a boat in the combustion tube kept at 1350° and pass the combustion gas into the absorber soln. (25 ml of 0·0003 *M* $K_2Cr_2O_7$ and 100 ml of 4 *N* HCl per litre) (5 ml); 10 min. after the termination of combustion, mix the absorber soln. with diphenylcarbazide soln. (50 mg in 25 ml of ethanol) (0·5 ml) and make up to 10 ml. Measure the extinction at 540 mμ after 10 min. The sulphur content is determined by reference to calibration curves obtained by the analysis of a standard sample of similar composition.

II. Reaction between potassium dichromate and sulphurous acid. Akira Sakihama. *Ibid.*, 1957, **6** (8), 487–491.—This reaction was studied potentiometrically and photometrically in 0·01 to 3 *N* HCl and H_2SO_4 soln. Reduction of $Cr_2O_7^{2-}$ to Cr^{3+} proceeds quant. in an O-free soln. In a soln. equilibrated with atmospheric O, the yield of Cr^{3+} increases with acid concn. and temp., but remains <25%. The results confirm those obtained in Part I.

K. SAITO

1826. **Oxidimetric determination of sulphides by the use of standard potassium ferricyanide solution.** Mitsuo Kiboku (Fac. of Sci. and Tech., Kinki Univ., Fuse, Osaka). *Japan Analyst*, 1957, **6** (8), 491–493.—The oxidation of S^{2-} with $K_3Fe(CN)_6$ proceeds quant. in 0·01 to 0·1 *N* NaOH of pH 10 to 12·5 and in Na_2CO_3 or ammoniacal soln. of pH 10 to 12. In a less alkaline soln., the reaction is too slow to cause quant. oxidation. In a more conc. NaOH soln., $S_2O_3^{2-}$ and SO_3^{2-} are formed and give high results. In the optimum pH range, no interference results from SO_3^{2-} or $S_2O_3^{2-}$. The end-point is indicated by the decolorisation of sodium nitroprusside.

K. SAITO

1827. **Fluorimetric and colorimetric estimation of sulphide and cyanide by demasking reactions of palladium chelates.** J. S. Hanker, A. Gelberg and B. Witten (Chem. Warfare Lab., Army Chem. Center, Md., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 93–95.—The fluorimetric determination is based on the de-masking of 8-hydroxy-5-quinolinesulphonic acid from non-fluorescent potassium di-(5-sulphooxino)palladium^{II} (**I**) with cyanide or sulphide, and the co-ordination of the liberated **I** with Mg^{2+} to form a fluorescent chelate, which is a measure of the amount of cyanide or sulphide. As little as 0·02 μg of cyanide or 0·2 μg of sulphide per ml of soln. can be determined. In the colorimetric method, 8-hydroxy-7-iodo-5-quinolinesulphonic acid is de-masked from the yellow potassium di-(7-iodo-5-sulpho-oxino)palladium^{II} with cyanide or sulphide, and reacts with Fe^{3+} to give a blue - green chelate, which is measured at 650 mμ. This method is sensitive to 1 μg of sulphide or cyanide per ml, but thiocyanate, thiols and some disulphides interfere.

E. G. CUMMINS

1828. **Colorimetric determination of sulphur dioxide.** G. D. Patterson, jun., and M. G. Mellon. U.S. Pat. 2,785,959; date appl. 19.3.57.—Atmospheric or aq. SO_2 is determined with vanadate - silica gels. Activated silica gel is mixed with 10 times its vol. of satd. aq. ammonium vanadate (**I**) and evaporated at 60°. A measured vol. of air, drawn through a tube containing the gel, forms five bands of colour. The concn. of SO_2 is determined by colour comparison with a standard set of colours or by a photocell. Tetraethoxysilane or sodium vanadate can be used instead of **I**.

CHEM. ABSTR.

1829. **Simple method of sulphate micro-determination.** Fujio Egami and Noriko Takahashi (Univ., Chikusa-ku, Nagoya, Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (5), 442–444.—Neutralised sample soln. (6 ml), containing 10 to 300 μg of S as SO_4^{2-}, and 1 ml of 0·01 *M* $BaCl_2$ are placed in a 10-ml flask. After 1 hr., 2 ml of 20% Na acetate is added, and the flask is cooled in ice for 20 min. One ml of 0·005 *M* $K_2Cr_2O_7$ is added and the soln. is made up to 10 ml and kept cold for 20 min. The mixture is centrifuged, and 2 ml of clear supernatant liquor are mixed with 1 ml of *N* NaOH and made up to 10 ml. The excess of chromate is determined spectrophotometrically in a 1-cm cell at 375 mμ. The result is read from the linear calibration curve obtained with known sulphate soln. Large amounts of phosphate interfere. The error is normally <5%. The method is suitable for sulphuric esters and for rain-water, but with the latter the sample size is increased to 100 ml and the liquid is concentrated after adding the $BaCl_2$.

A. B. DENSHAM

1830. **Stilbnaphthazo as an indicator for the volumetric determination of sulphate ions.** L. V. Markova. *Ukr. Khim. Zhur.*, 1957, **23** (1), 89–91; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,797.—4:4′-Di-(2-hydroxy-1-naphthylazo)stilbene-2:2′-disulphonic acid (stilbnaphthazo) (**I**) in neutral or weakly acid soln. in the presence of acetone (**II**) (2 pt. of **II** to each pt. of soln.) changes from pink to blue on the addition of barium salts. Sulphates can be titrated in the presence of **I** with soln. of barium salts. If **I** is used as a soln. in *iso*amyl alcohol (**III**), **II** can be dispensed with; the end-point is detected by the decolorisation of the **III** layer and by the appearance at the interface of blue films of the **I** - Ba^{2+} complex. Both with **II** and **III**, the determination of sulphates gives reproducible and satisfactorily accurate results. The error in determining 0·05 to 19 mg of SO_4^{2-} is ±2·4% in the presence of **II**, and ±2% in the presence of **III**.

C. D. KOPKIN

1831. **Potentiometric determination of sulphates.** N. A. Ugol'nikov and D. E. Galkin. *Uch. Zap. Tomskiĭ Univ.*, 1955, (26), 147–152; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,822.—The potentiometric determination of sulphates by means of titration with lead nitrate in aq. ethanolic soln. with a ferri-ferrocyanide indicator electrode was tested. The results show that, by comparison with the gravimetric determination of sulphates, this method is satisfactorily accurate and considerably more rapid. The possibility is indicated of determining sulphates potentiometrically by titration to an e.m.f. of zero. A non-compensation method of titration with a bimetallic system of electrodes is presented. C. D. KOPKIN

1832. [**Colorimetric**] **methods for analysis of dithionite.** E. F. Goebel and A. Roseira (Inst. Nac. de Tecn., Rio de Janeiro). *Rev. Quim. Ind., Rio de Janeiro*, 1957, **26**, 20–22.—Methods commonly used are subject to error from atmospheric oxidation of dithionite and are not adapted to the presence of interfering substances. By the use of Indanthrene yellow G (C.I. No. 1118), the amount of dithionite present induces a hypsochromic shift which may be measured by comparison with a colour scale (0·5 to 2 g of dye ≡ 3 to 6 g of $Na_2S_2O_4$ and 4·5 to 9 g of NaOH in the dye-bath). Another method is based on the decomposition of the dyes Azorubine (C.I. 179) and Amaranth (C.I. 184). Excess of dye is used and the amount of dithionite is derived from the colorimetric measurement of the soln. before and after reaction, by the use of a given formula. The methods are claimed to be quick and reliable. G. F. PENNY

1833. **Paper-chromatographic separation of inorganic anions containing sulphur.** Y. Servigne and C. Duval. *Compt. Rend.*, 1957, **245** (21), 1803–1804.—R_F values (ascending front) of S-containing anions (as sodium salts) on paper are as follows: (*a*) in *n*-butanol-water (3 + 1), SO_4^{2-}, $S_2O_3^{2-}$, $S_2O_6^{2-}$, $S_2O_5^{2-}$, S^{2-}, $S_2O_5^{2-}$ and HSO_4^-, 0·0; $S_4O_6^{2-}$ 0·02, SO_3^{2-} 0·03, $S_2O_8^{2-}$ 0·36, $S_2O_7^{2-}$ 0·57; and (*b*) in *iso*propyl alcohol-water (3 + 1), SO_4^{2-} 0·43, $S_2O_3^{2-}$ 0·35, $S_2O_6^{2-}$ 0·38, S^{2-} 0·32, $S_2O_5^{2-}$ 0·34, HSO_4^- 0·33, $S_4O_6^{2-}$ 0·39, SO_3^{2-} 0·11, $S_2O_8^{2-}$ 0·50, $S_2O_7^{2-}$ 0·51. The detection of $S_2O_5^{2-}$, SO_4^{2-}, $S_2O_6^{2-}$, HSO_4^-, $S_4O_6^{2-}$ and $S_2O_7^{2-}$ is by a benzidine-$KMnO_4$ reagent which gives a blue coloration, with a detection limit of 5 μg per 0·01 ml. Other ions are detected by standard procedures. D. A. PANTONY

1834. **Determination of tellurium in lead and lead alloys.** N. W. Fletcher and R. Wardle (The Chloride Electrical Storage Co. Ltd., Exide Works, Swinton, Manchester, England). *Analyst*, 1957, **82**, 743–746.—The sample is dissolved in a mixture of Br and HBr, excess of Br is removed by boiling and, when the temp. of the cooled liquid has fallen to 80°, $SnBr_2$ is added to precipitate the Te. After 1 hr. the ppt. is collected in a sintered-glass crucible, re-dissolved in a Br-HBr mixture and the soln. is carefully evaporated to reduce the vol. to a few ml. To the liquid are added HBr and ascorbic acid soln., the mixture is diluted to a known vol. and its extinction is measured at 442 mμ against a reagent blank and referred to a calibration graph. There is no interference from other alloying elements, and Se, if present, is volatilised during removal of the free Br. A. O. JONES

1835. **Oxidimetric titration with standard potassium ferricyanide solution. Determination of chromium, molybdenum and tungsten.** Mitsuo Kiboku (Fac. of Sci. and Technol., Kinki Univ., Fuse, Osaka). *Japan Analyst*, 1957, **6** (6), 356–359.—Chromium is reduced with zinc amalgam in 0·2 to 2·5 *N* HCl and titrated with $K_3Fe(CN)_6$ (**I**) in a basic soln. of pH < 10·7 in the presence of $Na_5P_3O_{10}$ (**II**). Sexavalent Mo is similarly reduced in 2 *N* H_2SO_4 and titrated with **I** at pH 9·1 to 11·2 in the presence of **II**. Tungsten is reduced in 4 *N* HCl and titrated at pH 6·6 to 10·6 in the presence of **II**. Sodium pyrophosphate can be used instead of **II** provided that the pH is kept slightly lower (≃0·3 pH unit). K. SAITO

1836. **Colorimetric determination of molybdenum.** A. M. Meshcheryakov. *Izv. Otd. Estestv. Nauk Akad. Nauk Tadzh.SSR*, 1956, (17), 3–16; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,811.—By using thiourea (**I**) instead of $SnCl_2$ as the reducing agent, the disadvantages of the thiocyanate method of determining Mo are eliminated, including fading of the orange colour with time and with increase of the quantity of $SnCl_2$. The colour formed is unchanged in 24 hr. and does not depend on the excess of **I**. Neutralise the soln. to litmus with NaOH soln. or aq. NH_3, add 20% NaCl soln. (25 ml), HCl (sp. gr. 1·16) (7 ml) or H_2SO_4 (sp.gr. 1·4) (5 ml), 1% soln. of $CuSO_4$ (1 ml), 5% soln. of **I** (5 ml) and 1 ml of a 50% soln. of NH_4SCN or a 64% soln. of KSCN, and dilute to 50 ml with water. After 20 min. compare the colour with that of a series of standard soln. If the colour is pale it is recommended to extract the coloured complex twice with *iso*amyl alcohol (3 ml and 2 ml). C. D. KOPKIN

1837. **Thermolysis of oxine molybdophosphate.** W. W. Wendlandt and J. A. Brabson (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 61–62.—Thermobalance experiments confirm that 140° is a good drying temp. for this ppt. The air-dried ppt. is converted into the anhydrous compound, $3C_9H_7ON.H_3(PMo_{12}O_{40})$, in the range 60° to 85° and this is stable over the range 85° to 285°. A decomposition product, stable between 335° and 375°, corresponds approx. to $C_9H_7ON.H_3(PMo_{12}O_{40})$, and at 470° this is converted into a stable oxide, $24MoO_3.P_2O_5$. E. G. CUMMINS

1838. **Rapid photometric determination of molybdenum in ferromolybdenum by the stannous chloride reduction method.** Shuichiro Mizoguchi (Toyo Denkyoku Kogyo Ltd., Hiratsuka, Shinagawa-ku, Tokyo). *Japan Analyst*, 1957, **6** (6), 376–381.—Experimental conditions, including the amount of H_2SO_4 and $SnCl_2$, the vol. of the sample soln. and the time of standing, were examined for the colorimetric determination of Mo at 470 mμ. Copper decreases the extinction but, in amounts >0·2 mg, to a definite extent. The extinction coeff. increases with increase in the amount of Fe, but remains constant in the presence of >5·0 mg. No interference results from V (<10 mg), W (<2 mg), Cr (<0·7 mg) or Ni (<10 mg). The time taken for an estimation is ≃20 min. *Procedure*—Heat the sample (1·0 g) with H_2SO_4 (1 + 1, 15 ml) and HNO_3 (2 ml) to white fumes, cool, and dissolve in water (80 ml). Add $CuSO_4$ (≃0·2 mg of Cu) and 10% $SnCl_2$ in HCl (1 + 1) (10 ml) and make up to 100 ml. Measure the extinction at 470 mμ after 2 min. K. SAITO

1839. **Indirect volumetric determination of molybdenum with ethylenediaminetetra-acetic acid.** E. Lassner and H. Schlesinger (Metallwerke Plansee G.m.b.H., Reutte, Austria). *Z. anal. Chem.*, 1957, **158** (3), 195–198.—The alkaline molybdate soln. is acidified with HCl, heated to boiling-point and made alkaline with aq. NH_3. The Mo is pptd. as calcium molybdate by the addition of $CaCl_2$ to the boiling soln., Eriochrome black T is added to the cooled soln. and the excess of Ca is chelated by titration with EDTA (disodium salt). The calcium molybdate is then dissolved by boiling with a measured excess of EDTA and the unchanged EDTA is back-titrated with a standard soln. of Mg. The amount of EDTA consumed in dissolving the ppt. is equiv. to the Ca and hence to the Mo in the ppt. Determinations made on known samples show an error of $\pm 1\%$. Interfering elements such as Cu, Ni, Co and Hg are masked by the addition of KCN; Fe and Al are removed in the preparation of the sample soln., but interference by W cannot be eliminated. G. BURGER

1840. **Rapid spectrophotometric determination of submilligram quantities of uranium.** C. A. Francois (Dow Chemical Co., Pittsburg, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 50–54.—Good separation of U from contaminants is achieved by a single extraction with an approx. 10% v/v soln. of tributyl phosphate in *iso*octane from an aq. phase containing approx. 500 g of $Al(NO_3)_3.9H_2O$ per litre. The yellow uranyl dibenzoylmethane complex formed by direct addition of an aliquot of the extractant to an acetone - water soln. of dibenzoylmethane and pyridine is measured spectrophotometrically at 410 mμ after being set aside for 1 hr. Common cations, except thorium in a concn. exceeding 10 times that of the uranyl ion, do not interfere. E. G. CUMMINS

1841. **γ-Ray spectrum of fission products from slow neutron irradiation of uranium-235.** D. H. Peirson (A.E.R.E., Harwell, England). *Brit. J. Appl. Phys.*, 1955, **6**, 444–449.—The fission products from ^{235}U irradiated by slow neutrons have been analysed by a two-crystal γ-ray scintillation spectrometer (*cf. Anal. Abstr.*, 1954, **1**, 2308). The spectrum between 1 and 70 days after irradiation has been recorded. The gross spectrum varies with time and consists of two groups of lines around 0·2 and 0·7 meV, with a single line at 1·58 meV as the only important component above 1 meV. By measuring energy and decay rates, 17 activities, due to fission products of high yield, were identified. The spectrometer is described. CHEM. ABSTR.

1842. **Luminescence determination of small quantities of uranium in igneous rocks.** L. L. Leonova. *Geokhimiya*, 1956, (8), 47–54; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,813.—A method is described for the determination of small quantities of U with complete decomposition of the rock (by soda fusion), separation of U from all accompanying elements as $U(HPO_4)_2.nH_2O$ on a zirconium carrier, and determination of the luminescence of NaF - U beads on a fluorimeter. Under the conditions described, the accuracy of the determination is from ± 5 to $\pm 10\%$, with a sensitivity of 5×10^{-6} % of U. C. D. KOPKIN

1843. **Micro volumetric determination of uranium with application to bismuth-base alloy analysis.** G. W. C. Milner and G. A. Barnett (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1957, **17** (2), 220–225 (in English).—Uranium in bismuth alloys is determined on the micro scale by reduction on a lead column, followed by oxidation with Ce^{4+}. After dissolving the sample (1 g) in HNO_3, Bi is eliminated as the bromide by evaporation with Br and HBr. Uranium is separated from Fe (previously reduced with SO_2) by adsorption on a column of De-Acidite FF resin (chloride form). The U is eluted with HCl, and an aliquot is evaporated to dryness and the organic matter destroyed by ignition. The U_3O_8 residue is dissolved in H_2SO_4, the soln. is passed through the micro lead reductor, and titrated with Ce^{4+}. Zirconium in 10-fold excess and Th in 100-fold excess do not interfere. J. H. WATON

1844. **Spectrochemical determination of impurities in uranium.** D. S. Pešić, V. M. Vukanović, S. N. Marinković and M. D. Marinković (Inst. Nucl. Sci. "Boris Kidrich," Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 71–77 (in English).—Procedures are described for the qual. detection of B (limit of 0·05 p.p.m.), Cd (0·1 p.p.m.), Fe, Cr, Cu, V and Li (1 p.p.m.), Mn (2 p.p.m.), Mo (3 p.p.m.) and Ni (5 p.p.m.); for the semi-quant. determination of V, Mo, Mn, Fe, Cr, Cu, Ni and Li; and for the quant. determination of B and Cd in the concn. range 0·5 to 20 p.p.m., with coeff. of variation of $\pm 7\%$ for B and $\pm 11\%$ for Cd. Samples of U_3O_8 (50 mg), obtained by ignition of uranyl nitrate in platinum crucibles at 1000°, were finely ground and mixed with spectrographically pure Ga_2O_3 (2 mg) as carrier, and transferred to cup-shaped graphite electrodes. For the quant. determination of B, Au (as $AuCl_3$ soln.) was used as internal standard, with the line pair B 2496·78 - Au 2675·95; for Cd, the line Cd 2288·02 was used, with no internal standard. J. H. W. FORSYTHE

1845. **Use of paper chromatography for the detection and determination of microgram amounts of inorganic fluoride.** R. J. Hall (Agric. Res. Council, Inst. of Animal Physiology, Babraham, Cambs., England). *Analyst*, 1957, **82**, 663–667.—To detect F^-, a $CaCl_2$ - $Th(NO_3)_4$ soln. is applied to chromatographic paper, which is then air-dried. Spots of 0·005 to 0·02 ml of the sample soln. containing 1 to 20 μg of F^- are then applied and the air-dried paper is developed in the descending-solvent manner with an acetone - acetic acid - water mixture. The paper is rinsed with water, immersed in aq. NH_3, rinsed again with water, drained and stained with Solochrome brilliant blue BS, when F^- appear as bright violet - blue spots. The excess of stain is washed out with 0·1 *N* H_2BO_3 soln. The spots are then stable and 1 μg of F^- can be detected. To determine F^-, a chromatogram is prepared, developed and dried as before. An area of the paper is cut out at the point of application, placed in 2 ml of 0·1 *N* HCl, which is then heated to b.p. After removal of the paper, enough 0·04 *N* NaOH is added to bring the pH to 2·9 to 3·0 (a method of attaining this critical value is described), and the F^- are then titrated with $Th(NO_3)_4$ soln. in the presence of a buffered alizarin red S soln. A procedure for removing interfering PO_4^{3-} and oxalate from the sample soln. is described. Recoveries of 89% were obtained for amounts of $F^- < 20$ μg. A. O. JONES

1846. **Rapid thermochemical method for determining active chlorine.** S. S. Shraĭbman (All-Union Sci. Res. Inst. of Reagents). *Zavod. Lab.*, 1957, **23** (8), 918–919.—The method is based on measurement of the temp. rise when the sample (a hypochlorite soln.) is mixed with $Na_2S_2O_3$ soln. G. S. SMITH

1847. **Indicator for chloride titrations.** F. E. Clarke. U.S. Pat. 2,784,064; date appl. 20.7.55.—A mixture of 2 g of diphenylcarbazone and 0·5 g of bromophenol blue per 100 ml of ethanol is used as an internal indicator for the titration of Cl^- with $Hg(NO_3)_2$. The pH is adjusted to 3·3 or to the yellow colour of the bromophenol blue before titration with $Hg(NO_3)_2$; a blue-violet colour is formed at the end-point. The indicator is stable for at least 6 months. CHEM. ABSTR.

1848. **Automatic titration of micro amounts of chloride by convection amperometry.** A. L. Juliard (Houdry Process Corp., Linwood, Pa., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 136–140.—The titration is carried out with a pre-treated silver cathode and a mercury anode polarised at 0·1 V. The end-point is reached when the detector current becomes minimum. Amounts of chloride ranging from 50 to 1000 μg in 30 ml of soln. containing from 0·1 to 2 g of other electrolytes can be determined in less than 5 min. with a standard deviation of ±10 μg. K. A. PROCTOR

1849. **The application of precipitation exchange reactions in analytical chemistry. IV. Ion-exchange reactions.** L. Erdey, É. Bányai and F. Paulik (Inst. allg. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1958, **13** (3–4), 453–463.—The exchange reaction between Cl^- and $Hg(IO_3)_2$ is discussed. Between certain limits of concn. the reaction $Hg(IO_3)_2 + 2\,Cl^- \rightleftharpoons HgCl_2 + 2IO_3^-$ proceeds without any side reactions. The determination of Br^-, CN^- and I^- may be carried out similarly. J. S. HETMAN

1850. **Rapid determination of manganese oxide in glass.** V. M. Yasinenko. *Steklo i Keramika*, 1957, (4), 22–23; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,852.—The proposed method enables MnO to be determined without treatment of the sample of glass with HF. After fusion and removal of the SiO_2 by double evaporation, make up the filtrate and wash liquors to 500 ml for a 2-g sample of glass or to 250 ml for a 1-g sample. To 100 ml of the soln. add H_2SO_4 (1 : 1) (20 ml) and evaporate to a vol. of 0·5 to 1 ml; Cl^- must be completely removed. Dilute the residue to 120 or 150 ml with hot water, filter and wash the filter two or three times with hot water. Heat the filtrate to boiling and add 10 ml of a freshly prepared $(NH_4)_2S_2O_8$ soln. Boil for an hour, filter off the ppt. with a dense filter and wash it eight or ten times to remove SO_4^{2-}. Dry the filter and ppt. in the funnel, transfer to a porcelain crucible, ash carefully, ignite and weigh as Mn_3O_4. C. D. KOPKIN

1851. **Use of electrolytically generated permanganate ion for coulometric titration.** O. A. Songina, N. G. Kemeleva and M. T. Kozlovskiĭ (Kazakh State Univ.). *Zavod. Lab.*, 1957, **23** (8), 896–900.—The coulometric determination of Fe^{II} in microgram amounts is carried out by titration with MnO_4^- electrolytically generated from 0·06 *M* $MnSO_4$ in 7 *N* H_2SO_4, and establishment of the end-point amperometrically. G. S. SMITH

1852. **Chelatometry. XIV. The ferric thiocyanate indicator.** P. Wehber and W. Johannsen (Lab. Metallhütte, Mark A.-G., Hamburg-Wilhelmsburg, Germany). *Z. anal. Chem.*, 1957, **158** (1), 7–9.—Ferric iron can be titrated at 20° with 0·1 *M* EDTA at pH 3·0 to 3·5 with thiocyanate as indicator. In the presence of a high concn. of acetate ions, the result is independent of thiocyanate concn. *Procedure*—To the strongly acid soln. (80 ml) add sodium acetate trihydrate (3 g) and enough chloroacetic acid to bring the pH to about 3 (bromophenol blue). Add 0·1% aq. Eriochrome green B (3 to 9 drops) and NH_4SCN (3 g), and titrate with 0·1 *M* EDTA. The colour changes from red to green at the end-point. A. R. ROGERS

1853. **Ultra-violet spectrophotometric determination of iron with ethylenediaminetetra-acetic acid.** Yasumitsu Uzumasa, Masakichi Nishimura and Toshiko Seo (Hokkaido Univ., Sapporo, Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (5), 438–442.—It is shown that Fe^{3+} can be determined spectrophotometrically with EDTA. Beer's law is obeyed up to 17 p.p.m. of Fe, in 0·1 *M* HCl, 0·001 *M* with respect to EDTA, at 260 mμ. The sensitivity is 0·0065 μg of Fe per ml. The extinction decreases in the light but recovers in the dark. The complex is stable in acid media, but the extinction decreases below pH 0·5 and in alkaline media. Nitric acid absorbs in the same region so that its concn. must be kept below 0·001 *M*. Acetic acid, H_2SO_4 or $HClO_4$ can be used; Cu^{2+}, Hg^{2+} and Ti^{4+} interfere, but other cations examined do not. A. B. DENSHAM

1854. **Ultra-violet spectrophotometric determination of iron(III) as a chloro complex.** Masayoshi Ishibashi, Tsunenobu Shigematsu, Yuuroku Yamamoto, Masayuki Tabushi and Toyokichi Kitagawa (Kyoto Univ., Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (5), 433–437.—The absorption spectra of aq. soln. of $Fe(ClO_4)_3$ containing various concn. of HCl are recorded. The results are interpreted in terms of the stepwise conversion of the aquo-ferric ion to $FeCl^{2+}$, $FeCl_2^+$, $FeCl_3$ and $FeCl_4^-$. At 340 mμ in 3 *N* HCl, Beer's law holds over the range 0·2 to 20 p.p.m. of Fe, but the sensitivity is low. Better results are obtained in the range 45 to 60 p.p.m. of Fe with 40 p.p.m. of Fe in the reference soln. The extinction does not vary significantly between 10° and 25°. There is interference from Cu^{2+}, Ti^{4+}, Bi^{3+} and V^{5+}, but none from Na^+, K^+, NH_4^+, Co^{2+}, Zn^{2+}, Mn^{2+} and Al^{3+} in concn. of 200 p.p.m. To determine Fe in ferrous alloys, 200 mg of sample is dissolved in 4 *M* $HClO_4$ on a sand bath. After cooling, several drops of 30% H_2O_2 are added and the soln. is evaporated almost to dryness. The evaporation is repeated several times, and the residue is made up with H_2O to 500 ml. An aliquot of 20 ml is mixed with 50 ml of 6 *M* HCl and made up with H_2O to 100 ml. The extinction of this soln. is measured at 340 mμ, with 40 p.p.m. of Fe in 3 *N* HCl as reference soln. With iron, haematite and ferromanganese the error based on single determinations is about ±0·4%, but +27% with ferrovanadium. A. B. DENSHAM

1855. **Spectrophotometric determination of iron with ethylenediaminedi-(o-hydroxyphenylacetic acid).** A. L. Underwood (Emory Univ., Ga., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 44–47.—This compound forms a chelate with Fe^{3+} giving a red colour. The reagent soln., which is stable for seven days, is prepared by suspending 1·8 g of solid in 50 ml of H_2O, and adding 6 *M* NaOH dropwise, with stirring, until a stable pH of 8·5 to 9·0 is reached, when most of the solid is dissolved. The volume is then adjusted to 100 ml and the suspension is filtered. A sample soln. (25 ml), containing the Fe (≃150 μg), 2 ml of reagent and 5 ml of buffer (60 ml of glacial acetic acid in H_2O, adjusted to pH 5·0 with NaOH, and diluted to 1 litre with H_2O), is measured

at 470 mμ and the iron content calculated from a standard curve. Constant absorption values are obtained in the pH range 2·5 to 8·8 and with the volume of reagent varying from 0·2 to 4·0 ml, although further excess of reagent does absorb at 470 mμ. The addition of buffer before reagent can cause up to one hour's delay in full colour development. Minimum errors are obtained in the range 3·2 to 8·0 p.p.m. of Fe, and over the range 2 to 10 p.p.m. the coeff. of variation does not exceed 1·1%. In the analysis of a soln. containing 150 μg of Fe, 100 mg of Pb, Al, Mn, Ca or Mg has no effect. At the level of 10 mg per 150 μg of Fe, Hg has no effect, while U, Th and Ni have a slight effect. At the level of 1 mg per 150 μg of Fe, U, Th, Cu, Ni and Zn have no effect, but Cr interferes. Interference from Co and Cr is persistent, even at low levels. E. G. CUMMINS

1856. **New quantitative spectrochemical precision method without internal standard. Application to iron.** A. Rodríguez Pérez. *Inst. Hierro y Acero*, 1957, **10**, 166–170.—The experimental technique for the spectrochemical determination of Fe is described. Intensity - concentration relationships for many of the characteristic Fe lines have been examined, some of which give parallel graphs. The most suitable lines for different amounts of Fe are indicated. L. A. O'NEILL

1857. **Analysis of high-purity iron.** R. E. Heffelfinger, D. L. Chase, G. W. P. Rengstorff and W. M. Henry (Battelle Memorial Inst., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 112–114.—This survey of the methods used for the determination of impurities includes details of separation of metallic impurities. Spectrographic analysis of the aq. soln. obtained by an ether extraction enables Mn, Cu, Ni, Cr, V, Co, Al, Pb, Ti, W and Zr to be determined. Elements in the sulphide group are pptd. and then determined spectrographically. Direct arcing of an HCl soln. of the sample is used for Si, Mg and Ca. Boron is determined spectrographically. Carbon is determined by a combustion method, and S and P are determined spectrophotometrically. Oxygen, H and N are determined by vacuum-fusion analysis. E. G. CUMMINS

1858. **Phase analysis of iron - nickel - titanium alloys.** M. M. Shapiro and G. E. Levit-Gurevich (Central Sci. Res. Inst. of Ferrous Metall.). *Zavod. Lab.*, 1957, **23** (8), 904–905.—The effect of temp. on the separation by electrolysis of the intermetallic phases in iron - nickel - titanium alloys, with various contents of Ti, is described. G. S. SMITH

1859. **Spectrographic determination of iron and manganese in titanium metal.** Yoshichika Oto and Chujo Matsumoto (Ind. Res. Inst., Osaka Prefecture, Enokojima, Nishi-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (6), 689–691.—Synchronised spark lines (with Feussner's generator, 10,500 V, 0·08 mH, 0·033 μF, exposure 180 or 45 sec.) of Fe and Mg in H_2SO_4 soln. of titanium are recorded with a quartz spectrograph, and Fe 2599·4 A and Mg 2795·5 A are compared with Ti 2572·7 A and 2841·9 A, respectively. The working curve is linear for 0·06 to 0·6% of Fe and Mg. The use of a porous cup carbon electrode (thickness of the bottom, 0·75 mm) is recommended. Traces of Mg in the carbon are removed by a d.c. arc discharge. The standard deviation is ≃8% for Fe and ≃1·8% for Mg. The sample (2 g for Fe and 5 g for Mg) is dissolved in 25% H_2SO_4 on a water bath, oxidised with HNO_3, mixed with tartaric acid (20 g for Fe, 30 g for Mg) and diluted to 100 or 250 ml before being introduced into the porous cup-electrode. K. SAITO

1860. **The rapid and routine analysis of iron-pickling liquor. III. Rapid determination of ferric nitrate in a hydrofluoric - nitric acid pickling liquor and some observations on the state of the iron.** Kimio Mukaewaki (Nippon Tokushu Steel Tube Co., Funato, Itabashi, Tokyo). *Japan Analyst*, 1957, **6** (7), 418–422.—In a hydrofluoric - nitric acid pickling liquor, Fe^{III} is present as the nitrate and a fluoro complex. This complex is stable in the pickling liquor and fails to oxidise KI. The amount of liberated iodine is regarded as corresponding to free Fe^{3+} [*i.e.*, $Fe(NO_3)_3$] and can be a measure of the potency of the liquor. The filtered sample-liquor (10 ml) is kept in thermostatically controlled apparatus regulated at the temp. at which the liquor is normally used, mixed with KI (5 g), shaken for 1 min., diluted with water and titrated with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. In the presence of nitric oxide, urea (0·2 g) is added. The accuracy is within ±5%. K. SAITO

1861. **Phase analysis of chrome - nickel - titanium steel with inter-metallide strengthening.** N. I. Blok, N. F. Lashko, K. P. Sorokina and F. F. Khimushin. *Zavod. Lab.*, 1957, **23** (8), 901–903.—Methods of electrolytic phase separation of EI-696 steel, *e.g.*, of composition C 0·05%, Ni 19·45%, Ti 2·53%, Cr 11·65%, Al 0·85% and B 0·02%, are described. G. S. SMITH

1862. **Analysis of carbides extracted from steel.** J. D. Hobson (Hadfields Ltd., East Hecla Works, Sheffield, England). *Analyst*, 1957, **82**, 708–710.—Investigations extending over several years have shown that the totals of the constituents of the analyses of carbide residues extracted from creep-resistant and precipitation-hardening steels failed to reach 100%. Evidence from examination of the residues by electron microscopy, from analysis and from investigations of the effects of changes of electrolyte suggested that the presence of O in the residues caused the discrepancy, although this was not confirmed by X-ray diffraction examination. Determination of the O present by a modification of the vacuum-fusion method (Cook *et al.*, *Anal. Abstr.*, 1954, **1**, 2302) confirmed that the failure to reach 100% in the chemical analysis was due to the presence of O and the discrepancy was thus quant. accounted for. A. O. JONES

1863. **Comparative evaluation of methods of determining gases in steel.** Yu. A. Klyachko, L. L. Kunin and E. M. Chistyakova (Central Sci. Res. Inst. of Ferrous Metall.). *Zavod. Lab.*, 1957, **23** (8), 905–907.—Two sets of apparatus (available in the USSR) for vacuum-fusion work are compared. G. S. SMITH

1864. **Development of a coincidence spectrometer and a method for analysis of cobalt-60.** H. R. Helmholz (Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. HW-49718, 1957, 67 pp.—A specific instrumental method is described for the determination of ^{60}Co in fission product waste streams. A combination of coincidence counting and energy discrimination is used to determine selectively only the high-energy photons arising in the decay of ^{60}Co. The procedure requires little chemical separation and is limited only by the total activity of the accompanying radionuclides. It is possible to determine 1000 disintegrations per min. to an

accuracy within ±6% in the presence of 10^5 pulses per min. arising in the detector due to impurities having either lower energy coincidences or higher energy single photons. The coincidence spectrometer is described in detail. N. E.

1865. **Rapid photometric determination of a large amount of cobalt in cobalt naphthenate.** Kōhei Shinoda, Tarō Noguchi and Shigeyuki Yamada (Nagoya Plant, Toa Gosei Chem. Ind., Minato-ku). *Japan Analyst*, 1957, **6** (7), 435–438.—This rapid (≃20 min.) determination of Co (10 to 250 mg) is effected by the measurement of the extinction at 517 mμ of an aq. soln. containing H_2SO_4 and H_3PO_4. The extinction remains unchanged in 1 to 5 *N* H_2SO_4 soln. The addition of 10 ml of H_3PO_4 (1:3) suffices for the masking of Fe^{3+} (< twice the weight of the Co). The presence of Ni (<0·6 times the weight of the Co), Cu (<0·4 times) and Mn (<2 times) does not cause interference under the given conditions. The results agree with those by the gravimetric method with 1-nitroso-2-naphthol. *Procedure*—Boil the aq. soln. of the sample containing 100 to 200 mg of Co with H_2SO_4 (1:1, 15 ml) and water (≃20 ml) for 10 min. Cool, and remove the oily substance by extraction with CCl_4 (50 ml). Boil to remove CCl_4, make up to 100 ml with H_3PO_4 (1:3, 10 ml) and measure the extinction.

K. Saito

1866. **New gravimetric method for the determination of cobalt in the presence of nickel and other elements.** T. I. Pirtea and E. Antonescu. *Rev. Chim., Bucharest*, 1957, **8** (9), 591–594.—This method is based on the conversion of the Co^{2+} into $[Co(NO_2)_6]^{3-}$, which are pptd. by $[Co(NH_3)_6]Cl_3$ (**I**) as $[Co(NH_3)_6][Co(NO_2)_6]$. The cryst. yellow ppt. settles quickly, is washed with reagent **I** (0·1%), ethanol and ether, dried in a vacuum desiccator and weighed. The authors claim the method to be rapid (30 to 35 min.), and that Ni can be determined in the filtrate by pptn. with dimethylglyoxime. Cobalt can be determined in the presence of Ni^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mn^{2+}, Zn^{2+}, Cu^{2+}, Pb^{2+}, Hg^{2+} and Cd^{2+}, but K^+ and NH_4^+ interfere. H. Sher

1867. **Determination of cobalt [in steel] with acridine.** Shigeo Wakamatsu (Toto Seiko Co., Minamisuna-machi, Koto-ku, Tokyo). *Japan Analyst*, 1957, **6** (7), 426–430.—Acridine (**I**) precipitates Co^{2+} in an HCl soln. containing a large amount of NH_4SCN (≃20 g for <0·1 g of Co). The pptn. is complete in >*N* HCl and at >20°. The ppt. is sol. in dil. HCl and must be washed with 0·5 *N* HCl containing ≃2% NH_4SCN. The voluminous ppt., after being washed, is dissolved in acetone (20 to 60 ml) containing ammonium acetate (50%, 10 ml), mixed with 50% NH_4SCN and titrated with 0·03 *M* EDTA. Ferrous iron and V^{4+} are not pptd. under the given conditions, and the ppt. formed by Mo and Zn are insol. in acetone. Copper (>0·3 mg) interferes. *Procedure*—Magnet steel (0·5 g) is decomposed with aqua regia, the WO_3 is filtered off and the filtrate is made up to 250 ml. A 50-ml portion is treated with $SnCl_2$ and diluted to 150 ml with H_3PO_4 (10 ml) to prevent the pptn. of Sn. The HCl concn. is made 0·5 to 1·0 *N* and the soln. is treated with NH_4SCN (50%, 40 ml) and **I** in ethanol (5%, 10 ml). The ppt. is washed, dissolved and titrated as described above. High-speed steel (W 18%, Cr 4%, V 1%, Mo 1%) (1 g) is dissolved in HCl (1:1), oxidised with HNO_3 and filtered. The filtrate is shaken with ether to remove most of the Fe and Mo, reduced with $SnCl_2$, made up to 250 ml with H_3PO_4 (10 ml) and treated similarly. In the presence of Cu, CuS is pptd. with H_2S; Fe^{III} is thus reduced to Fe^{II}. K. Saito

1868. **Studies on the platinum group. X. The alkali fusion of osmium and ruthenium and its application to their determination.** Kiyoshi Watanabe (Oizumi Mfg. Co. Ltd., Nerima-ku, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (7), 983–986.—Alkaline soln. of OsO_4 have two absorption maxima, at 250 and 295 mμ; the extinction coeff. in the visible region, *e.g.*, at 520 mμ, is proportional to concn. of Os of $<1·5 \times 10^{-5}$ *M*. When Os is fused with Na_2O_2 and NaOH at 600° for 1 hr., the aq. extract shows the same absorption. It is likely that very weakly bonded $HOsO_4^-$ are produced in the soln. When these aq. soln. are reduced with ethanol, Na_2OsO_4 is produced quant., which has a max. absorption at 520 mμ, the extinction being proportional to concn. of Os of <0·015 *M*. The compound Na_2RuO_4, with two absorption max. at 390 and 350 mμ, is less stable than Na_2OsO_4, and cannot be used for colorimetric determinations. A mixture of Os and Ru is fused with Na_2O_2 and NaOH, and the aq. extract is heated with ethanol at 30° to 40° for 15 min.; Os is determined colorimetrically in the supernatant soln. The black ppt. of Ru is dissolved in conc. HCl, diluted to 4 *N* and determined colorimetrically with thiourea. Only a negligible amount of Os is found in the ppt.

K. Saito

1869. **Electro-chromatography in the separation of ions. II. Separation of precious metals.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta). *Anal. Chim. Acta*, 1957, **17** (2), 228–231 (in English).—The behaviour of Pt^{4+}, Os^{4+}, Ir^{4+}, Ru^{3+}, Rh^{3+}, Au^{3+} and Pd^{2+} on a strip of Whatman No. 2 filter-paper (44 cm × 1 cm), when kept at a potential of 150 V for 5 hr., has been studied with a variety of electrolytes. The results show that quaternary or ternary mixtures may be separated into distinct zones of the individual ions.

J. H. Waton

1870. **Paper-electrochromatographic separation of the platinum-group metals.** M. L. Dunton (Ohio State Univ., Columbus, U.S.A.). *Dissert. Abstr.*, 1957, **17** (5), 970.—Separations of Pt, Pd, Rh and Ir have been achieved by the horizontal paper-strip electrochromatography of their complexes with EDTA. The various factors influencing electromigration rates, namely pH, concn. of reagent, and ageing of metal soln., were studied. Complete separation of the four metals is possible when a two-fold molar excess of reagent at pH 9 is used. Under these conditions, the rate of migration to the anode increased in the order Pd, Pt, Ir; Rh is pptd. at the point of application of the sample. The results of these electromigration studies were also applied to the continuous electrochromatographic separation of the platinum-group metals. G. S. Roberts

1871. **Systematic analysis of silicates by the use of ion-exchange resin.** Jun Yoshimura and Hirohiko Waki (Fac. of Sci., Kyushu Univ., Hakozaki, Fukuoka). *Japan Analyst*, 1957, **6** (6), 362–369.—The separation of Fe (total), P, Al, Ti, Mn, Ca, Mg, Na and K in silicate rocks is described. The sample (0·1 g) is decomposed by the method of Sweet *et al.* (*Brit. Abstr. C*, 1952, 473), oxidised (Fe^{II} to Fe^{III}) with H_2O_2, acidified to give 15 ml of 10 *N* HCl soln., and passed through column I [Dowex 1-X8(Cl)]. This resin is eluted first with 10·5 *N* HCl to give an eluate (*A*) containing Na, K,

Mg, Ca and Al, then with 6 *N* HCl to give an eluate (*B*) containing Mn, Ti and P, and finally with 0·5 *N* HCl to give an eluate (*C*) containing Fe. Eluate *A* is passed through a column of Amberlite IR-120 (H); Na and K are eluted, respectively, with 0·2 *N* HCl and 0·5 *N* HCl; and Mg, Ca and Al with 4 *N* HCl. This last fraction is diluted to 0·2 *N* HCl and passed through a column of Dowex 50-X12(NH_4^+), which is eluted with *M* and 2*M* ammonium acetate to liberate Mg and Ca, respectively, and with 4 *N* HCl to liberate Al. Eluate *B* is passed through a column of Amberlite IR-120(H), which is washed to remove P, then eluted with 0·8 *N* and 1·8 *N* H_2SO_4 to separate Ti and Mn. The various metal ions are determined by usual methods. The results for quartz, diabase, andesites and obsidian are in good agreement with those obtained by standard methods. K. SAITO

1872. **Rapid method of analysis of blast-furnace slag.** P. I. Protsenko (Kushvinsk Metall. Works). *Zavod. Lab.*, 1957, **23** (8), 911–912.—Rapid methods of sufficient accuracy are described for determining SiO_2, Al_2O_3, MnO, CaO, MgO and FeO. G. S. SMITH

See also Abstracts—**1757**, Fast grey RA as indicator in EDTA titrations. **1769**, Determination of molybdenum in rocks. **1891**, Determination of iron in adipic acid. **1943**, Determination of copper in blood, urine and tissue. **1944**, Determination of arsenic in biological products. **1979**, Complexometric determination of bismuth. **1981**, Determination of cerium in pharmaceutical preparations. **2010**, Determination of uranium in natural waters. **2013**, Determination of ^{89}Sr, ^{90}Sr, ^{137}Cs and ^{144}Ce in water. **2015**, Determination of antimony, barium and cadmium in trade effluents. **2020**, Carrier precipitation of trace elements. **2021**, Chromatographic separation of valency states of metals. **2022**, Determination of aluminium in soil extracts. **2024**, Spectrophotometric analysis of iron chelates. **2035**, Density - composition tables for aqueous solutions of nitric acid. **2065**, Determination of gases in uranium.

3.—ORGANIC ANALYSIS

1873. **Simplified determination of ^{14}C-containing compounds.** H. Götte, R. Kretz and H. Baddenhausen (Max Planck Inst. f. Chem., Mainz, Germany). *Angew. Chem.*, 1957, **69** (17), 561–562.—*Procedure*—Fuse a platinum wire into the ground glass stopper of a 500-ml conical flask. To the wire attach a platinum gauze to hold the sample. Pack the accurately weighed sample (0·1 to 100 mg) in an ashless filter or, if in soln., absorb on the paper and dry. Alternatively, inject the soln. into a cotton-wool pellet. Attach a narrow strip of filter-paper to serve as a fuse. Place 50 ml of *N* NaOH in the flask and fill the flask with oxygen. Immediately ignite the fuse and insert the stopper in the flask so that neither the gauze nor the strip touches the NaOH. Grasp the stopper to counteract the pressure caused by the rapid combustion. After cooling, shake the flask to ensure quant. absorption of CO_2 in the NaOH. Precipitate $BaCO_3$ as usual in the presence of NH_4Cl. Measure the activity in a counter. Satisfactory results are shown for ^{14}C-labelled oxalic acid and for poly(vinylpyrrolidone). The method is also suitable for radio paper chromatography, the strip being cut out and burned as described above. It is not suitable for substances with low specific activity because extraneous CO_2 from the paper reduces the activity. D. F. PHILLIPS

1874. **Micro-colorimetric determination of organic sulphur by the tin(II) - strong phosphoric acid reduction method.** Toshiyasu Kiba, Ikuko Akaza and Sachiko Taki (Univ., Kanazawa, Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (5), 482–486.—The sample, solid or soln., containing 1 to 35 μg of S, is evaporated to dryness with 0·5 ml of 1% $BaCl_2$ soln. in a silica test-tube. The residue is treated with 1 ml of Cr^{VI} - H_3PO_4 reagent, and heated until the colour changes to green, showing that oxidation is complete. After cooling and adding 5 ml of Sn^{II} - H_3PO_4 reagent, a glass cap carrying inlet and outlet tubes is fitted, and an absorption tube containing 35 ml of 2% $ZnSO_4.2H_2O$ soln. is connected. The air is swept out with purified CO_2, the CO_2 rate is reduced to 1 bubble per sec., and the silica tube is heated for 15 min. after white fumes appear. After a further 5 min. the absorption vessel is disconnected and placed in a thermostat at 24°. After a few minutes, 1·5 ml of *p*-aminodimethylaniline soln. [0·7% in H_2SO_4 (1:1)] is added, followed immediately after shaking by 0·5 ml of 10% $FeCl_3.6H_2O$ soln. The stoppered tube is kept at 24° for 15 min., the soln. is made up to 50 ml, and the extinction is measured within 30 min. at 655 mμ in a 1-cm cell. The Cr^{VI} - H_3PO_4 reagent is prepared by dissolving 12 g of $K_2Cr_2O_7$ in 100 g of conc. H_3PO_4, purified by heating to 300° with 1% of $SnCl_2.2H_2O$. The Sn^{II} - H_3PO_4 reagent is prepared by heating 50 g of $SnCl_2.2H_2O$ with 250 g of conc. H_3PO_4 to 300°. The CO_2 gas is washed in turn with water, alkaline $KMnO_4$, VSO_4, $BaCl_2$ and Zn acetate. The calibration curve is obtained with Na_2SO_4 soln. For several org. compounds, recoveries of S of over 98% are obtained with samples containing <6 μg of S. A. B. DENSHAM

1875. **Analysis for industry. [Determination of other elements in fluorinated organic compounds.]** A. M. G. Macdonald. *Ind. Chem. Mfr*, 1957, **33**, 360–362.—A review is presented of the literature on the micro-determination of C, H, N, Cl, Br, I, S, P and As in fluorinated org. substances, and miscellaneous polarographic, mass-spectrometric and gas-chromatographic procedures. (29 references.) S.C.I. ABSTR.

1876. **Rapid semi-micro determination of acetyl and methyl groups attached to carbon. I. The titration and distillation of acetic acid.** Takero Sudo, Daizo Shimoe and Takatomo Tsujii (Kurosaki Factory, Mitsubishi Chem. Ind., Kurosaki, Yawata). *Japan Analyst*, 1957, **6** (8), 494–498.—Kuhn's method for the determination of acetyl groups (*Ber.*, 1933, **66**, 1274) was examined with reference to the distillation and titration of acetic acid. For a rapid determination, the distillate is first neutralised only approx. with 0·02 *N* NaOH, then boiled with 0·01 *N* HCl (1 ml) for 20 sec. and titrated accurately with 0·02 *N* NaOH in a hot soln. to phenolphthalein. The still was slightly modified; the amount of H_2SO_4 (1 + 2) must be <3 ml and that of Na_2SO_4 >5 g for 10 ml of the hydrolysed soln. The steam-distillation is complete within 15 min. for <40 mg of sample. The error is $<0·2\%$ (relative) for $\simeq 10$ mg of acetic acid.

II. Determination of acetyl and C-methyl groups. Takero Sudo, Daizo Shimoe and Takatomo Tsujii. *Ibid.*, 1957, **6** (8), 498–502.—The hydrolysis of O- and N-acetyl groups was re-examined with reference to increase of the temp. above 200°. At 240° the

reaction is complete within 30 min. (for both O- and N-acetyl groups) in the presence of 3 ml of H_2SO_4 (1 + 2) for <40 mg of the sample. The modified still can also be used for the determination of C-methyl groups by the Kuhn method (*loc. cit.*). The chromic acid must be recrystallised from 80% H_2SO_4. The sample (<40 mg) is heated with a mixture (8 to 10 ml) of H_2SO_4 and 5 *M* aq. CrO_3 soln. (1 + 4, by vol.) for 30 min. (occasionally 90 min.). The excess of CrO_3 is reduced with 30% H_2O_2, and steam-distillation is carried out in the presence of Na_2SO_4 (8 g). Volatile samples are heated in a micro Carius tube. K. SAITO

1877. **Identification of alkylthio groups by infra-red spectroscopy.** A. Menefee, D. O. Alford and C. B. Scott (Union Oil Co., Brea, Calif.). *J. Org. Chem.*, 1957, **22** (7), 792–795.—A number of identifying absorption bands have been assigned to the following groups: methylthio, ethylthio, *n*-propylthio, *iso*propylthio and *n*-butylthio. N. E.

1878. **Rapid method for the determination of low concentrations of ethylene by means of a detecting tube.** Yoshitaka Kobayashi (Nat. Univ., Yokohama). *Yûki Gôsei Kagaku Kyôkai Shi*, 1956, **14**, 137–141.—Silica gel, on which is absorbed a mixed soln. of ammonium molybdate and palladium sulphate, is packed in a tube and used for the detection of ethylene in air. The light-yellow colour changes to dark blue. The concn. of ethylene in air is determined by passing the air through at constant speed for a certain time and measuring the length of the dark-blue layer and comparing with standards. CHEM. ABSTR.

1879. **Determination of moisture in gases containing ethylenic hydrocarbons.** F. M. Rapoport, P. A. Khodak and T. I. Shatrovskaya. *Trudy Gos. Nauch. i Proektn. Inst. Azotn. Prom.*, 1956, (5), 364–367; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,864.—The titrimetric method described consists in absorbing the moisture in ethanediol by passing the gas through it at a rate of 20 litres per hour and titrating the solution obtained by the Karl Fischer method. The reproducibility of results is satisfactory, the difference between parallel determinations being $>2{\cdot}4\%$, and on the average 1·5%. C. D. KOPKIN

1880. **Carbon tetrachloride.** British Standards Institution (2 Park St., London). B.S. 575:1957, 10 pp.—In this revised Standard, a change has been made in the sp. gr. limits, a reduction has been made in the limit for sulphur compounds, and no limit is specified for water. Methods are described for the determination of distillation range, residue on evaporation, and sulphur compounds, and for the detection of acidity and free chlorine. O. M. WHITTON

1881. **Diethyl ether (technical).** British Standards Institution (2 Park St., London). B.S. 579:1957, 11 pp.—In this revised Standard, sp. gr. limits at 25°/25° have been introduced for use in tropical climates, and a limit for water (Karl Fischer determination) has been set. Methods are described for the determination of distillation range, residue on evaporation, and acidity, and for the detection of peroxides and sulphur compounds. O. M. WHITTON

1882. **Colorimetric estimation of chloral (hydrate).** O. P. Malhotra and V. D. Anand (Hindu Univ., Benares). *J. Indian Chem. Soc.*, 1957, **34** (7), 501–504.—The Fujiwara colour reaction has been used for the determination of chloral hydrate. Beer's law is obeyed. *Procedure*—A mixture of 40% NaOH soln. (5 ml), colourless technical pyridine bases (10 ml) and acetone (2 ml) is added to the test soln. (5 ml) containing 2·4 mg of chloral hydrate. The mixture is heated on a boiling-water bath for 5 min., then cooled to 20° and the pyridine layer is separated and diluted with acetone (to 20 ml). The colour can be matched against standards in a Lovibond Tintometer. For very small amounts of chloral hydrate, the use of pure rather than technical pyridine is recommended. C. A. SLATER

1883. **A new technique for the chromatographic separation of organic compounds.** R. Sargent and W. Rieman, III (Rutgers Univ., New Brunswick, N.J.). *J. Org. Chem.*, 1956, **21**, 594–595.—The separation of non-electrolytes is greatly improved by using a salt soln. instead of water as the eluent. Diethylene glycol and dipropylene glycol can be quant. separated on a 10-cm column of Dowex 1-X8, sulphate form, by eluting with 3 *M* $(NH_4)_2SO_4$. The separation of methanol, ethanol and *n*-propanol is also improved by the use of the same eluent. N. E.

1884. **Determination of methanol and methyl acetate in vinyl acetate by infra-red spectroscopy.** Yutaka Nishino and Tsugio Kominami (Toyama Plant, Kurashiki Rayon Co., Toyama). *Japan Analyst*, 1957, **6** (8), 507–510.—Methanol (**I**) in vinyl acetate (**II**) (0·05 to 2%, by vol.) is determined spectrophotometrically in a 0·1-mm cell by the use of its absorption at 2·78 μ. The extinction of the absorption peak of methyl acetate (**III**) at 3·37 μ is proportional to the concn. of **III** (0·2 to 5%, by vol.). **I** also has an absorption at 3·37 μ, which obeys Beer's law for concn. of $<2{\cdot}0\%$ (by vol.) and the result for **III** must be corrected for the **I** content. The error is $\simeq \pm 0{\cdot}03\%$ for $<2\%$ of **I** and $\simeq \pm 0{\cdot}2\%$ for $<6\%$ of **III**. Water must be removed from **II** with K_2CO_3. The time taken for an estimation is $\simeq 3$ hr., including the time for dehydration. K. SAITO

1885. **Method for the identification of pentitols and hexitols.** J. Baddiley, J. G. Buchanan and B. Carss (King's Coll., Univ. of Durham, England). *J. Chem. Soc.*, 1957, 4138–4139.—By treatment with 2 *N* HCl at 100° for 17 hr., pentitols and hexitols are converted in part into anhydro compounds. The products, after evaporation, can be separated by ascending paper chromatography in *n*-propanol - aq. NH_3 (sp. gr. 0·88) - water (6:3:1). Unchanged polyols and anhydro compounds can be detected by spraying the paper with the periodate - Schiff reagents for glycols (Buchanan *et al.*, *J. Chem. Soc.*, 1950, 3162). A table of R_F values for the polyols and for the coloured spots is presented. All the pentitols and hexitols can be identified, the number and colour of the spots being characteristic in each case and readily observed with samples of 100 μg of the original polyol. G. BURGER

1886. **Formaldehyde solution.** British Standards Institution (2 Park St., London). B.S. 2942:1957, 13 pp.—Methods are given for the determination of ash, acidity, iron, aldehyde content and methanol content of formaldehyde (for use in plastics and chemical manufacture generally) and the necessary amounts or limits are specified. O. M. WHITTON

1887. **Estimation of formaldehyde by Romijn's method in presence of ammonium salts.** S. Bose (Dept. of Chem., Mahakoshal Mahavidyalaya,

Jabalpur, India). *J. Indian Chem. Soc.*, 1957, **34** (7), 497–500.—It has been found that a mixture of 3% $HgCl_2$ and 40% KI prevents the interference of ammonium salts in the determination of formaldehyde. *Procedure*—Formaldehyde soln. (≃0·1 *M*, 5 ml) is treated with 8 ml of a freshly prepared soln. of 3 g of $HgCl_2$ and 50 g of KI in 100 ml of H_2O, and 12·5 ml of 0·1 *N* iodine is added, followed by 2 ml of *N* NaOH. The mixture is cooled and set aside for 20 min. at 20°, then acidified with *N* HCl (4 ml) and titrated against 0·05 *N* $Na_2S_2O_3$. A blank is run. The accuracy of the method is within ±0·25%. C. A. SLATER

1888. **Paraformaldehyde.** British Standards Institution (2 Park St., London). B.S. 2941:1957, 10 pp.—Methods are given for the determination of ash, water-insoluble matter, acidity, iron and aldehyde content of paraformaldehyde (for use in plastics and chemical manufacture generally) and limits are specified for each case. O. M. WHITTON

1889. **Solid derivatives of aldehydes. II. A specific reagent for aldehydes: 1:2-di-(*p*-chlorobenzylamino)ethane.** J. H. Billman, Ju-Yu Chen Ho and L. R. Casswell (Dept. Chem., Indiana Univ., Bloomington, U.S.A.). *J. Org. Chem.*, 1957, **22** (5), 538–539.—Almost all of the common aldehydes form 2-substituted 1:3-di-(*p*-chlorobenzyl)iminazolines with 1:2-di-(*p*-chlorobenzylamino)ethane. A list of 26 compounds so formed and their m.p. is given. N. E.

1890. **Simplified procedure for preparing α- and β-elaeostearic acids and a revised spectrophotometric procedure for their determination.** J. S. Hoffmann, R. T. O'Connor, D. C. Heinzelman and W. G. Bickford (S. Reg. Res. Lab., New Orleans, La., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (7), 338–341.—Equations for the calculation of either α- or β-elaeostearic acid or total elaeostearic acid content from u.v. absorption data (*cf.* O'Connor *et al.*, *J. Amer. Oil Chem. Soc.*, 1947, **24**, 212) have been revised. G. HARDING

1891. **Determination of parts per million of iron in adipic acid by ultra-violet absorption.** R. E. Seeber, R. G. White and K. H. Ferber (National Aniline Div., Allied Chemical and Dye Corp., Buffalo, N.Y.). *Appl. Spectroscopy*, 1957, **11** (3), 113–114.—The absorption band at 360 mμ, due to the $FeCl_3$ complex, is used as a measure of the iron content of adipic acid. The solvent is 15% (v/v) conc. HCl in 95% ethanol with the addition of H_2O_2 to ensure that the Fe is present in the ferric state. This solvent has the advantage over aq. soln. in that close adherence to Beer's law is attained, and the extinction of the $FeCl_3$ complex is independent of Cl^- concn. A 10-g sample is dissolved to make 100 ml of soln. and a 100-mm absorption cell is used to cover the range 0·25 to 15 p.p.m. with a precision of ±0·25 p.p.m. P. T. BEALE

1892. **The micro-determination of tartaric acid.** K. Luthardt and R. Pohloudek-Fabini (Pharm.-Chem. Inst., Univ., Greifswald, Germany). *Pharm. Zentralh.*, 1957, **96** (8–9), 453–457.—Two methods are described, both of which are applicable to samples eluted from paper chromatograms. (*i*) To 4 ml of an aq. soln. or eluate (containing 20 to 90 μg of acid) ammonium metavanadate soln. (0·5 ml of 5% soln.) and water to 5 ml are added. After 10 min. the extinction is read against water. (*ii*) The aq. soln. or eluate (20 to 100 μg of acid) is evaporated to dryness in a vacuum oven, the residue is dissolved in 2 ml of a freshly prepared *p*-dimethylaminobenzaldehyde soln. (4% in dry freshly distilled acetic anhydride) and heated to 140° ± 2° for 10 min. After cooling to room temp., acetic anhydride is added to 5 ml and the extinction is determined. D. P. FELIX

1893. **Resolution of acid mixtures in non-aqueous solvents. Potentiometric titration of dibasic acids with quaternary ammonium titrants.** G. A. Harlow and G. E. A. Wyld (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 69–72.—The influence of the solvent on the resolution of dibasic acids by potentiometric titration was studied. Curves are presented for the titration of H_2SO_4, oxalic and succinic acids in mixtures of H_2O and *iso*propyl alcohol and for H_2SO_4 in *iso*propyl alcohol, ethanol, methanol, diethyl ether and pyridine. Mixtures containing acids of different charge type, which cannot be resolved in water, can be readily resolved in non-aqueous solvents. G. P. COOK

1894. **Quantitative estimation of halogenated organic acids on paper chromatograms.** M. H. Hashmi (West Reg. Lab., C.S.I.R., Lahore, Pakistan). *Anal. Chim. Acta*, 1957, **17** (2), 225–227 (in English).—Bromo and iodo acids are determined to an accuracy within 2% after chromatographic separation. The acids are applied to a Whatman No. 4 filter-paper as a soln. in dil. aq. NH_3, and the chromatogram is developed with propanol - conc. aq. NH_3 (7:3). After location with the help of a standard chromatogram, the paper containing the ammonium salts is cut out and heated under reflux with metallic sodium and abs. ethanol. The soln. is diluted with H_2O, acidified with HNO_3, and titrated with $AgNO_3$ soln., eosin and di-iododimethylfluorescein being used as indicators for the bromo and iodo acids, respectively. J. H. WATON

1895. **A rapid polarographic method for the determination of the double-bond content in maleic acid polyesters.** J. Voigt (Dtsch. Akad. Wiss., Berlin-Adlershof). *Plaste u. Kautsch.*, 1957, **4**, 3–6.—The polyester is saponified by adding aq. alkali to an acetone soln. of the ester. The maleic and fumaric components can be rapidly determined polarographically with a max. error of ±3%. The following polyester types were investigated: maleic acid (**I**) - ethylene glycol (**II**); **I** - hexamethylene glycol (**III**); **I** - diethylene glycol; **I** - adipic acid - **II**; **I** - phthalic acid - **II**; fumaric acid (**IV**) - **II**; **IV** - **III**; and the partially cross-linked product of **I** - **II** polyester plus 100 ml % of styrene plus 1% of benzoyl peroxide. The isomerisation of maleic acid to fumaric acid as found in the experiments is discussed. CHEM. ABSTR.

1896. **The separation of fatty esters and aldehydes by gas - liquid chromatography.** J. C. Hawke, W. L. Dunkley and C. N. Hooker (Fats Research Lab., D.S.I.R., Wellington, N. Zealand). *N.Z. J. Sci. Tech., B*, 1957, **38** (9), 925–938.—The construction of a relatively simple chromatographic apparatus and thermal conductivity detector is described. Quant. determinations of normal and branched-chain fatty acid esters up to C_{20} are made by measuring recorded peak areas. Mixtures of lower aldehydes are resolved by gas - liquid chromatography. I. JONES

1897. **Di-*n*-butyl phthalate.** British Standards Institution (2 Park St., London). B.S. 573:1957, 9 pp.—In this revised Standard, the limits for colour

and colour stability have been reduced, sp. gr. limits at 25°/25° have been introduced for use in tropical climates, and a limit for water (Karl Fischer determination) has been set. Methods for the determination of colour stability, ash, volatile matter, acidity, and ester content of di-*n*-butyl phthalate are given. O. M. WHITTON

1898. **Analysis of polyethylene glycol esters.** J. D. Malkemus and J. D. Swan (Jefferson Chem. Co. Inc., Austin, Texas, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (7), 342–344.—The analysis of mixtures of mono-ester, di-ester and polyethylene glycol, which may result from the esterification of polyethylene glycols with fatty acids, or from the reaction between ethylene oxide and fatty acids, is based on the extractability of the glycol with water. Three extractions with satd. NaCl soln. at 95° to 100° ensure complete removal of the glycol. The organic ester-containing layer is then dried under vacuum and filtered, and the hydroxyl and saponification values are determined. By assuming that the mol. wt. of the fatty acid and polyethylene glycol are known, the composition of the mixture can be calculated from given equations. G. HARDING

1899. **Determination of acrylonitrile [vinyl cyanide] vapour by means of detector tubes.** Yoshitaka Kobayashi (Nat. Univ., Yokohama). *Yûki Gôsei Kagaku Kyôkai Shi*, 1956, **14**, 673–675.—Silica gel (60 to 80 mesh) (100 g) is treated with a soln. of conc. H_2SO_4 (25 ml), H_2CrO_4 (10 g) and H_2O (100 ml), and dried *in vacuo* at 100°; 0·2 g is sealed in a glass tube (length of layer 60 to 80 mm). The sample gas (100 ml) is blown through the tube in 200 min. and, from the length of the dark-green layer, the amount of vinyl cyanide (**I**) is calculated. The measuring range is 1 to 120 mg of **I** in 1 litre of air. Reducing substances interfere.

CHEM. ABSTR.

1900. **Potentiometric determination of amides in acetic anhydride.** D. C. Wimer (Abbott Lab., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 77–80.—Amides in acetic anhydride soln. can be titrated potentiometrically by $HClO_4$ in acetic acid or dioxan. Data for ≃40 amides are presented. The results are discussed in relation to the probable equilibria involved and evidence for an increased acidity function is presented and a reaction mechanism postulated. Certain amides cannot be titrated and these are discussed with regard to configuration. G. P. COOK

1901. **Tritolyl phosphate.** British Standards Institution (2 Park St., London). B.S. 1999: 1957, 10 pp.—In this revised Standard, the clause governing the *o*-tolyl ester content has been deleted, and the limits for sp. gr. and viscosity have been changed, and that for acidity lowered. Methods are given for the heat treatment for colour stability, and the determination of volatile matter and acidity. Limit tests for water-soluble impurities are described. O. M. WHITTON

1902. **Ultra-violet spectroscopic analysis of alkylbenzenes.** K.-D. Ledwoch (Rheinpreussen A.-G. für Bergbau und Chem., Homberg, Germany). *Erdöl u. Kohle*, 1957, **10** (9), 595–596.—A study of the u.v. spectra (*K* 1605 band) of alkylbenzenes establishes a linear relationship between the Bunsen extinction coeff. α and the number of carbon atoms in the alkyl residue, namely, $y = -0{\cdot}6x_{1605K} + 113{\cdot}25$ (for $I_0 = 100\%$ transmission), where y is the alkyl fraction (%) of the alkylbenzene. The molar extinction coeff. ϵ being const. for the series, namely, 20·25 ($I_0 = 100\%$), measurement of density and mol. wt. will provide sufficient data for a complete analysis of a mixture. J. L. PROSSER

1903. ***o*-Dichlorobenzene (Grades A and B).** British Standards Institution (2 Park St., London). B.S. 2943–4: 1957, 14 pp.—Specifications are given for the sp. gr., distillation range, crystallising point, ash, acidity, and free chlorine of *o*-dichlorobenzene of Grade A (substantially free from other isomers) and of Grade B (normally containing much *p*-dichlorobenzene), respectively. Methods for the determination of distillation range, crystallising point, and ash, and for the detection of acidity and free chlorine are given. O. M. WHITTON

1904. **Determination of phenols in phenol-containing material by high-frequency titration.** B. P. Ershov, V. L. Pokrovskaya, V. A. Zarinskiĭ and D. I. Koshkin. *Khim. Prom.*, 1957, (2), 106–107; *Ref. Zhur., Khim.*, 1957, Abstr. No. 60,901.—Phenols are determined with the apparatus described earlier (Zarinskiĭ and Koshkin, *Anal. Abstr.*, 1954, **1**, 2275). In carrying out the determination, treat up to 5 g of the phenol-containing material with excess of a 10% aq. alkali soln., make up to 500 ml and dilute 0·25 to 5 ml of the soln. to 50 ml for high-frequency titration with HCl. The beginning and end of the decomposition of the phenoxides are determined by the position of the horizontal section of the graph of μA *vs.* vol. of added HCl. The method may be used for the titration of pure phenol, technical phenol, a mixture of the three isomers of cresol, and technical cresol. The accuracy of the method is within ±1·5%. The time for the analysis is 10 to 15 min. C. D. KOPKIN

1905. ***p*-Phenylazobenzoyl chloride for identification and chromatographic separation of colourless compounds. III. Phenols.** E. O. Woolfolk and J. M. Taylor (Central State Coll., Wilberforce, Ohio). *J. Org. Chem.*, 1957, **22** (7), 827–829.—The brilliantly coloured aryl-*p*-phenylazobenzoates have been found to be suitable derivatives for separation of mixtures of phenols by chromatographic adsorption. A list of 30 phenols and the m.p. of their *p*-phenylazobenzoates is given.

N. E.

1906. **Polarographic determination of quinol in methyl methacrylate.** V. N. Dmitrieva and V. D. Bezuglyĭ (Kharkhov Works for Dental Materials). *Zavod. Lab.*, 1957, **23** (8), 914–915.—The sample (6 ml) containing 0·001 to 0·1% of quinol is dissolved in 9 ml of methanol and 5 ml of water and mixed with 0·4 g of NH_4NO_3. After removal of O, the quinol wave ($E_{\frac{1}{2}} = +0{\cdot}23$ V *vs.* the S.C.E.) is recorded and measured. The method of addition is used to determine the amount of quinol.

G. S. SMITH

1907. **Colorimetric determination of vanillin.** N. S. Kapur, K. M. Narayanan, G. S. Bains and D. S. Bhatia (Central Food Technol. Res. Inst., Mysore, India). *Chem. & Ind.*, 1957, (38), 1272–1273.—Thiobarbituric acid reacts with vanillin to give a stable yellow complex in the presence of H_3PO_4, this complex having a max. absorption at 432 to 434 mμ. As little as 0·2 μg per ml of vanillin in 92% H_3PO_4 gives a colour with 0·025 *M* thiobarbituric acid in *M* H_3PO_4. The method is applicable to the determination of other aldehydes.

H. B. HEATH

1908. **Semi-micro Kjeldahl procedures for pyridinium halide and oxyhalide salts.** V. B. Fish and P. R. Collier (Lehigh Univ., Bethlehem, Pa., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 151–152.—The semi-micro Kjeldahl method gives excellent recovery (>98%) of N from a wide variety of pyridinium halide and oxyhalide salts. The use of tin-foil sample-cups is convenient and the resulting tin salts in the digestion mixture increase the recovery of ammoniacal N. The method is applicable to both refractory and non-refractory material.
G. P. Cook

1909. **Simultaneous determination of thiophen or its homologues in mixtures with unsaturated hydrocarbons.** B. P. Fedorov and G. I. Gorushkina. *Khim. i Tekhnol., Topliva*, 1957, (12), 5–12; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,846.—It is established that Picon's method for the quant. determination of thiophen or its homologues by acetylation with acetyl chloride in the presence of $SnCl_4$, with subsequent conversion of the ketones into the oximes, does not give accurate results if there are unsaturated hydrocarbons present in the mixture. A bromide - bromate method is evolved for the determination of thiophen, 2- or 3-methylthiophen, and 2:5-dimethylthiophen in binary mixtures with unsatd. hydrocarbons. For determining the composition of ternary mixtures containing thiophen, or 2- or 3-methylthiophen, benzene or toluene and unsatd. hydrocarbons, the bromide - bromate method is also recommended, in conjunction with the lamp method for determining S. To determine unsatd. hydrocarbons in mixtures containing 2:5-dimethylthiophen and xylene the Hübl - Waller iodine method is recommended.
C. D. Kopkin

1910. **Determination of acenaphthylene in mixtures of acenaphthylene and acenaphthene.** L. S. Soloveĭchik and A. P. Nikolaeva (Inst. of Polymerisation Plastics). *Zavod. Lab.*, 1957, **23** (8), 916.—The material (≃0·3 g) is dissolved in 20 to 25 ml of CCl_4, then mixed with 4 g of mercuric acetate and 30 ml of acid- and aldehyde-free methanol and heated (>50°) to complete dissolution of the mercuric acetate. After 12 to 15 min., 75 ml of neutral satd. NaCl soln. and 70 to 80 ml of water are added and the soln. is titrated with 0·1 *N* NaOH to the phenolphthalein end-point. A blank is carried out at the same time. The titration measures the amount of acetic acid set free as a result of the reaction at the ethylene double bond of acenaphthylene.
G. S. Smith

1911. **Detection of pyrene, benzo[α]pyrene, and other polynuclear hydrocarbons.** E. Sawicki and R. R. Miller (U.S. Dept. Health, Educ. and Welfare, Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 109–110.—Solutions of pyrene and benzo[α]-pyrene in dimethylformamide gave a brilliant dark-blue colour when treated with aq. tetraethylammonium hydroxide soln. The sensitivities were 5 and 15 μg, respectively. Eight other polynuclear hydrocarbons gave colours varying from red to violet and about 15 other hydrocarbons gave essentially negative reactions. The test was applied to hydrocarbon, fluorenone and quinone samples.
G. P. Cook

1912. **Fluorimetric determination of 1- and 2-naphthol in mixtures.** D. M. Hercules and L. B. Rogers (M.I.T., Cambridge, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 96–99.—The determination of 1- and 2-naphthol is by measurement of the fluorescence in 0·1 *M* NaOH at 480 and 426 mμ, respectively. The fluorescence is excited by 365-mμ radiation. The precision and accuracy are within ±4% in the concn. range of 5 to 120 μ*M* for 23 determinations on synthetic mixtures. Naphthalene, 2-chloronaphthalene and naphthalene-2-sulphonic acid did not interfere in 30-fold excess, and the presence of O, small changes in temp. and moderate exposure to u.v. radiation did not cause significant errors.
G. P. Cook

1913. **Selectivity of various liquid substrates used in gas chromatography.** H. M. Tenney (Esso Standard Oil Co., Baton Rouge, La., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 2–8.—Eighteen different liquid substrates were tested for their selectivity towards types of hydrocarbons and oxygenated compounds. For hydrocarbons, highly selective materials are available for the separation of aromatics from saturated compounds, and cyclic hydrocarbons can be separated from acyclic compounds in a mixture having a b.p. range of 20° to 25°. More efficient substrates are needed to separate acyclic olefins from equivalent boiling naphthenes, and diolefins from acyclic olefins. Separation of ethers from other oxygenated types (except acetals) appears to be feasible for a mixture of boiling range of 80° to 90°. Alcohols may be separated from other types over a boiling range of 15°. Ketones are reasonably well separated from esters but not from aldehydes. The dipropionitriles have the highest selectivity of those compounds tested, for both hydrocarbon and oxygenated compound types.
G. P. Cook

1914. **Determination of five- to seven-carbon saturates by gas chromatography.** F. T. Eggertsen and S. Groennings (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 20–25.—A blend of 25 C_5 to C_7 saturates can be analysed for all components, except 3-ethylpentane, by gas chromatography with three different types of column. The average accuracy was 0·13% and the average deviation was about 0·3% absolute. In general, the results agreed with those obtained by an A.P.I. co-operative testing of the same sample by other procedures. Mass spectrometry was utilised to study interferences by C_8 hydrocarbons which partially overlap the C_7's; these can be corrected for by the application of a combined gas chromatographic - mass spectrometric procedure.
G. P. Cook

1915. **Gas chromatography of olefins. Determination of pentenes and hexenes in gasoline.** H. S. Knight (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 9–15.—Gas-chromatographic techniques are described for the determination of C_5 and C_6 olefins. Fifteen C_5 olefins, including the six mono-olefins, *cyclo*pentene, *cyclo*pentadiene and the chain dienes, and the seventeen possible C_6 mono-olefins, except for the pair 3- and 4-methylpent-1-ene, can be resolved. The pentene and hexene fractions from a catalytically cracked petrol contained all but one or two of the possible mono-olefins and more cyclic olefins than dienes. Analysis of known blends gave errors of < ±2%. Detection was by thermal conductivity.
G. P. Cook

1916. **Testing liquid combustible materials. Determination of content of water and impurities in heating and raw oils. DIN 51793.** Fachausschuss Mineralöl- u. Brennstoffnormung des FNM. *Erdöl u. Kohle*, 1957, **10** (9), 616–617.—The oil (50 ml) is

thinned with 50 ml of benzene and placed in a centrifuge tube, the tube is stoppered, shaken, maintained at 60° for 10 min., and centrifuged at 1200 to 2000 r.p.m. (according to the dimensions of the tube) for 10-min. periods until the vol. of ppt. remains constant. J. L. PROSSER

1917. **Testing liquid combustible materials. Determination of content of non-settling water in heating oils. DIN 51786.** Fachausschuss Mineralöl- u. Brennstoffnormung des FNM. *Erdöl u. Kohle*, 1957, **10** (9), 615.—The oil (200 ml) is placed in a measuring cylinder for 24 hr., at 20° for a light oil, 50° for a middle oil, and 80° for a heavy oil, after which the water content of the upper 65% is measured by the method of DIN 51582. J. L. PROSSER

1918. **Combustion - amperometric titration of traces of halogen in petroleum products.** L. J. Cali, J. W. Loveland and D. G. Partikian (Sun Oil Co., Marcus Hook, Pa., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 74–77.—A Beckman atomiser burner is used for the determination of Cl in benzene, toluene and naphtha samples. The combustion is carried out in an enclosed system and the inorganic Cl formed is absorbed in aq. Na_2CO_3 soln. and titrated amperometrically with $AgNO_3$. The range of Cl studied was 1 to 10 p.p.m. The standard deviation of results from known values is $\pm 0{\cdot}6$ p.p.m., and the repeatability standard deviation is $\pm 0{\cdot}3$ p.p.m. G. P. COOK

1919. **Determining sulphur compounds in petroleum naphtha. The Humble scheme.** J. H. Karchmer (Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 80–85.—An integrated scheme for the determination of thiols, H_2S, elementary S, aliphatic and cyclic sulphides, disulphides, thiophens and total S is presented. The scheme outlines the features of seven principal methods and variations in which polarographic, potentiometric, spectrophotometric, mass-spectrometric and chemical techniques are employed. References to procedures, lists of special solvents, and other data are given. G. P. COOK

1920. **Determining disulphides in petroleum naphtha. Modification of the acetic acid - zinc reflux method.** J. H. Karchmer and M. T. Walker (Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 85–90.—An investigation of the low results sometimes obtained showed them to be due to the volatility and water solubility of certain thiols formed by reduction of the disulphides. These errors can be avoided by the use of an absorption trap, with nitrogen purging, to catch the low-mol.-wt. thiols, and by titration of the reflux bottoms without the separation of the acetic acid. Seventeen primary, secondary, tertiary and aromatic disulphides ranging from di-methyl to di-*tert.*-dodecyl disulphide were studied, and recoveries were generally >97% by the modified procedure for all except tertiary disulphides. G. P. COOK

1921. **Quantitative determination of organic disulphides.** R. L. Hubbard, W. E. Haines and J. S. Ball (U.S. Bureau Mines, Laramie, Wyo., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 91–93.—Three chemical methods and a polarographic procedure for the determination of disulphides in petroleum were tested with 15 disulphides of known purity. The chemical reduction procedures selected were: acid-reflux, acid-stirring, and alkali methods, the first two involving reduction with zinc and acetic acid at elevated and room temp., respectively. The acid-reflux method gave the best overall recoveries, but poor recovery of disulphides of lower mol. wt. The acid-stirring procedure gave good recoveries with all the normal and secondary disulphides, and the alkali procedure gave good results for only the normal compounds. Polarography is unsatisfactory for a wide range of disulphides. An acid-stirring method can be recommended for routine work when tertiary compounds are not involved. G. P. COOK

1922. **Simple method for the chromatographic analysis of lubricating oil fractions.** G. Schwarz (Österreichische Mineralölverwaltung A.-G., Reichsratstrasse 1, Vienna). *Erdöl u. Kohle*, 1957, **10** (9), 588–592.—Mineral oil is separated into aromatic-free, aromatic, and resinous fractions by adsorption on a silica-gel column from petroleum soln., followed by elution with, respectively, light petroleum, benzene, and acetone. Data are given on the change of structure resulting from refining or H_2SO_4 treatment, and on the distribution of S in the three components. J. L. PROSSER

1923. **Rapid chromatographic analysis of soap-thickened lubricating greases.** G. W. Powers, jun., and F. J. Piehl [Standard Oil Co. (Indiana), Whiting, U.S.A.]. *Anal. Chem.*, 1958, **30** (1), 28–31.—The grease is stirred with $CHCl_3$, and ethanesulphonic acid is added. The soln. is transferred directly to a column of silica gel impregnated with ethanolamine. Oil is eluted from the column with $CHCl_3$ and then fatty acids are eluted with 5% acetic acid in $CHCl_3$. The solvent is removed from the two eluates and the residues are weighed. Data from the analysis of oils, commercial acids, synthetic mixtures and commercial greases suggest that the method is accurate to within 0·5% by wt. of oil and 0·3% by wt. of acid for the majority of soap-thickened greases, including those prepared from ester and silicone oils. The results compared well with those obtained by ASTM methods. The average standard deviations for recoveries of oil and acid from replicate analyses of 3 commercial greases were $\pm 0{\cdot}26$ and $\pm 0{\cdot}12$% by wt., respectively. G. P. COOK

1924. **Determination of tributyl phosphate in kerosine solutions.** A. J. Fudge and G. C. Hutton (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2384, 1957, 18 pp.—A kerosine soln. containing $\simeq$40 mg of tributyl phosphate is hydrolysed by heating under reflux with 2 ml of 55% HI for 15 min. After cooling, 10 ml of H_2O and 3·5 ml of conc. HNO_3 are added through the condenser to decompose excess of HI, and when the reaction has subsided the condenser is removed, and the liquid is heated until colourless. After being cooled, the soln. is made up to 50 ml, and about 10 ml is filtered through a dry 541 filter-paper. A 5-ml aliquot of the filtrate is treated with a little water, 5 ml of HNO_3 (1:2), 5 ml of 0·25% ammonium vanadate and 5 ml of 5% ammonium molybdate, and made up to 50 ml. The extinction of the molybdovanadophosphate colour is read against a blank, in a Spekker photo-electric absorptiometer, with a mercury vapour lamp and H 503 and Ilford 601 filters, and results are referred to a standard curve prepared from soln. of K_2HPO_4 containing from 0·1 to 1·0 mg of P. Recovery was 99 to 100%. The final soln. should not contain more than 1·0 mg of P in 5 ml as, above this, Beer's law is not obeyed. R. E. ESSERY

1925. **Determination of anionic and non-ionic surface-active agents in aqueous solution.** Yuzi Takayama (Mitsubishi Rayon Co., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (7), 872–874.—Karush and Sonenberg's method for the determination of anionic surfactants (*A*) (*Brit. Abstr. C*, 1950, 171) was applied to a mixture of *A* and non-ionic surfactants (*N*), and found to be satisfactory if the wt. of *N* present was <30 times that of *A*. Stevenson's method for the analysis of *N* (*Anal. Abstr.*, 1954, **1**, 3038) can be satisfactorily applied in the absence of *A*, but is subject to interference by substances other than those mentioned by Stevenson. *Procedure*—Dilute the sample (0·03 to 0·8 mg of *N*) to 10 ml, add $BaCl_2$ (10%, 2 drops), warm at 50° for 5 min. and centrifuge (3500 r.p.m.) for 5 min. to separate *A*. Dissolve the residue in boiling water (5 ml), cool and centrifuge. Repeat this procedure three times, combine the supernatant liquors and make up to 25 ml. Then apply Stevenson's method.

K. Saito

1926. **Formulae for estimation of esters and alcoholic constituents in essential oils. VIII. Simple formulae and their applications.** J. B. Lal (H.B. Technol. Inst., Kanpur). *J. Instn Chem., India*, 1957, **29** (3), 150–154.—Formulae are devised and experimentally established for calculating the composition of mixtures containing two alcohols, an ester, an acid and a ketone. H. B. Heath

1927. **Simplified control analyses of solutions used in partial acetylation of cotton.** E. M. Buras, jun., A. S. Cooper and M. D. Cruz (S. Reg. Res. Lab., New Orleans, La., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 104–107.—Volumetric methods are described for the rapid analysis of mixtures of acetic acid, acetic anhydride and $HClO_4$, and of acetic acid, H_2O and $HClO_4$ which occur in the partial acetylation of cotton. In the determination of $HClO_4$ by titration in anhydrous acetic acid soln. with KH phthalate and 1-naphtholbenzein indicator, the error does not exceed $\pm 0{\cdot}1\%$ in the concn. range 0 to 10% of $HClO_4$. Water and acetic anhydride are determined empirically by phase separation in the presence of a large vol. of hydrocarbon solvent; a precision of $\simeq 5\%$ is obtained for both water and acetic anhydride. An alternative procedure for the determination of high concn. of anhydride ($>33\%$) involves the titration of the sample with a hydrocarbon solvent until a permanent cloudiness is obtained; the vol. of titrant is compared with a standard calibration curve.

G. P. Cook

1928. **The determination of water in cosmetic emulsions.** M. Zahradník and V. Pokorná (Pražské Kosmetické Závody, Prague). *Průmysl Potravin*, 1957, **8** (7), 371–373.—The sample (0·1 g) is polished in a very thin layer on a microscope slide and dried at 70° to const. weight. The method is suitable for samples containing $>20\%$ of H_2O, when the Karl Fischer method yields inaccurate results. J. Zýka

1929. **pH value of aqueous extracts of paper.** British Standards Institution (2 Park St., London). B.S. 2924: 1957, 11 pp.—In this Standard, the pH value of the paper refers to its acidity or alkalinity in terms of the hydrogen-ion concn. of the unfiltered aq. extracts of the paper. Both the cold extraction procedure, which gives results that correlate better with the back-water pH at which the paper was made, and the hot extraction method, which gives results that correlate with the tendency of the paper to inhibit the drying of printing inks, are described. The procedures deal with the pH of an extract of the whole paper. O. M. Whitton

1930. **Gravimetric estimation of medulla in wool.** D. A. Ross and J. B. Speakman (Univ. Leeds, England). *Text. Res. J.*, 1957, **27**, 345–351.—A gravimetric estimation of the amount of medulla in wool is made by determining the amount of residue left after samples of known dry weight have been treated with a 1·6% soln. of peracetic acid for 48 hr. at 25°, followed by *N* aq. NH_3 for 24 hr. at 25°. A correction must be applied for the amount of residue given by non-modulated wools, approx. the same for Merino and crossbred wools. As the amount of residue obtained from any wool after treatment with peracetic acid and aq. NH_3 increases with the temp. of drying and the regain from which it is dried, it is best that dry weights of samples which are to be used for analysis should not be determined directly, but calculated from the measured regains of samples weighed out at the same time as those used for analysis.

Chem. Abstr.

1931. **Rapid test for resin identification on textiles.** J. H. Howard (Cranston Print Works Co., R.I., U.S.A.). *Amer. Dyest. Rep.*, 1957, **46**, 313.—The scheme presented for identifying aminoplasts on coloured and uncoloured cotton fabrics is based on the removal of aminoplasts by acid hydrolysis. Urea - formaldehyde, ethyleneurea - formaldehyde and melamine - formaldehyde resins all have different acid hydrolysis rates. Formaldehyde liberated during the acid hydrolysis provides a means of identifying the respective resins through reaction with phenylhydrazine and $FeCl_3$, since colours characteristic of the individual resins are given. The liberation of NH_3 when an acid extract of the resin is boiled with NaOH indicates the presence of a urea - formaldehyde resin. E. S. Lane

1932. **The use of physico-chemical methods in black-liquor analysis.** R. Domanský (Chem. Inst. Acad. Sci., Bratislava, Czechoslovakia). *Papir a Celulosa*, 1957, **12** (7), 145–146.—A survey is presented, with a critical evaluation of the potentiometric determination of the alkalinity, the argentimetric determination of sulphides, and the flame-photometric determination of sodium.

J. Zýka

1933. **Infra-red analysis of a copolymer.** K. Ueberreiter and W. Krull (Fritz-Haber Inst., Berlin-Dahlem, Germany). *Angew. Chem.*, 1957, **69** (17), 557–561.—Quant. i.r. analysis of copolymers of styrene and acenaphthylene is described, use being made of the characteristic absorption bands at 6·87 μ and 12·22 μ, respectively. Alternative methods of preparing the samples are suggested—(*i*) by pouring a thin film of the benzene soln. of the polymer on to a mercury surface, (*ii*) by pressing the sample mixed with KBr into tablets. The styrene band is independent of the mutual arrangement of the chain members, whereas the acenaphthylene band is not. The intensity decrease and the simultaneous shifting of the acenaphthylene band are dependent on the styrene content of the copolymer. The results enable conclusions to be drawn as to the structure of the chain. D. F. Phillips

1934. **Use of adsorption chromatography in the analysis of rosin acids and hydroxy rosin acids.** V. A. Pentegova and A. I. Lisina. *Izv. Vost. Fil.*

Akad. Nauk, SSSR, 1957, (1), 65–69; *Ref. Zhur., Khim.*, 1957, Abstr. No. 63,818.—The separation of mixtures is carried out on a column of activated carbon (**I**), silica gel (**II**) or moist (6%) Al_2O_3. Add to the column a soln. of 0·1 g of the mixture in 2 or 3 ml of solvent and elute the first acid with the same solvent. Determine the completion of elution of the first acid with alkaline phenolphthalein soln. and proceed to the elution of the second acid, with a different solvent. The first acid analyses at $C_{20}H_{30}O_2$ and represents ≃90% of the acid content of the mixture. It is eluted from **I** by chloroform - butanol (9:1) and from **II** with benzene. To elute the second acid (analysing $C_{20}H_{30}O_4$) use benzene with some ethanol added. The oxidised products are separated from the rosin acids, being insoluble in light petroleum; they have a $[\alpha]_D$ of from +37·4° to +43·7° and m.p. >200°. On treatment with ethanol or acetone they separate into soluble and insoluble fractions. The main constituent of the former fraction is the dihydroxy acid $C_{20}H_{30}O_4$. The latter fraction is also insoluble in ether, pyridine and chloroform, but is readily soluble in a mixture of methanol and benzene (1:1), and less readily soluble in a mixture of ethanol and benzene, the $[\alpha]_D$ of the latter fraction (in the last-named solvent) being from +36·8° to +47·0°. The main component of the fraction insoluble in ethanol or acetone is the optically active tribasic acid approximating in composition to $C_{70}H_{122}O_7$. In admixture with this acid is a product of high mol. wt. and low acid value. C. D. KOPKIN

1935. **Progress in varnish testing.** J. C. Weaver (Sherwin Williams Co., Cleveland, U.S.A.). *Amer. Paint J.*, 1957, **41** (38), 82–90.—A progress report of the A.S.T.M. Varnish Subcommittee is given. The main matters considered are the effect of interfering substances, *e.g.*, dibasic acids and phenols, on the determination of rosin by selective esterification methods, and the standardisation of conditions for measuring the viscosity of varnishes by bubble-tube methods. L. A. O'NEILL

1936. **Detection of urea, melamine, *iso*cyanate and urethane resins. Rapid group test for nitrogen, silicon, phosphorus and titanium in coating materials.** M. H. Swann and G. G. Esposito (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 107–109.—*p*-Dimethylaminobenzaldehyde gives a yellow colour with –N:C:O groups and can be used for the detection of *iso*cyanate and urethane resins; the test is specific in the presence of acetic acid. When urea resins are refluxed in the presence of this reagent and of certain proportions of acetic acid and acetic anhydride (3:10), a bright-blue colour is developed. During this test, melamine (from melamine - formaldehyde resins) is detected by the formation of a white insoluble complex after the refluxing. The presence of N, Si, P and Ti in coating materials is detected by the usual colour tests after acid digestion of the sample with H_2SO_4 and $HClO_4$. G. P. COOK

1937. **The characteristics of crude montan wax and their evaluation for quality determination. II. Evaluation of the characteristics.** W. Schaack and D. Födisch (VEB Braunkohlenwerk "Gustav Sobottka," Röblingen-am-See). *Fette, Seif., Anstrichmitt.*, 1957, **59** (4), 209–213.—The quality of crude montan wax is dependent on the content of resin- and asphalt-like materials. The characteristics of artificial mixtures prepared from crude wax are determined (*cf. Anal. Abstr.*, 1957, **4**, 4053), and the results are used to establish methods for determining the proportion of asphalt, resin and pure montan wax in crude wax. The residue on ignition is directly proportional to the amount of asphalt in the sample; pure montan asphalt yields 3·30% of residue, and crude wax free from asphalt 0·04%. For crude waxes with 7 to 60% of resin, the proportion of matter insol. in acetone at 20° is directly related to the resin content. The viscosity at 90° and the amount of paraffin-insoluble matter can also be used to indicate the composition of crude wax. A nomogram is given to enable resin and asphalt contents to be determined rapidly from different characteristics. E. HAYES

1938. **Infra-red analysis of emulsion polishes.** J. E. Murphy and W. C. Schwemer (S. C. Johnson & Son, Inc., Racine, Wis., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 116–126.—A rapid method for the determination of wax, resin, polymer and emulsifier is described. The spectra of the common components of emulsion polishes and of a group of amine emulsifiers, as their hydrochloride derivatives, are presented. K. A. PROCTOR

1939. **Determination of free sulphur in acetone extracts [of rubber].** Wasaburo Jono, Teizo Watanabe, Kannosuke Miyaji and Hideo Konishi (Dept. of Applied Chem., Fac. of Engng, Kobe Univ., Nagata-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (6), 789–790.—The influence of various vulcanisation accelerators on the polarogram of S (*cf. Anal. Abstr.*, 1958, **5**, 814) was examined. No marked change was observed except that guanidine derivatives decreased the wave height. When pptd. $CaCO_3$, chalk, ZnO, $MgCO_3$ or carbon black is present in the rubber, the wave becomes less distinct. An acetone extract of vulcanised rubber containing <1% of an accelerator such as mercaptobenzothiazole, dibenzothiazolyl disulphide or tetramethylthiuram disulphide gives a two-step wave under the given conditions, but the first wave, which is independent of the amount of S, disappears on addition of gelatin. This wave appears to be the adsorption wave of S. The results of sulphur content in these vulcanised rubbers, except for those containing tetramethylthiuram disulphide, agree with results by the gravimetric method. K. SAITO

1940. **Qualitative test for nitroglycerin.** J. Lamond (Chem. Inspectorate, Station Approach Buildings, Kidbrooke, London). *Analyst*, 1957, **82**, 768–769.—In the test described, freshly prepared Nessler reagent is added to an equal vol. of an ethanolic extract of the sample. If the concn. of nitroglycerin in the ethanolic extract is not less than 1%, a black ppt. is rapidly produced. Other components of propellent explosives produce white opaque liquids or orange or red colours. A rough extraction of the finely ground sample (2 to 5 g) with aq. 80% ethanol (5 to 10 ml) effects sufficient segregation of the nitroglycerin to render the test specific. A. O. JONES

See also Abstracts—**1764**, Distribution of normal paraffins in waxes. **1803**, Determination of germanium in coal ash. **1812**, Determination of thiourea. **1980**, Determination of 7-oxocholesterol in wool wax. **1991**, Separation of food dyes.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1941. **A new polarographic procedure for measuring the blood oxygen tension *in vitro*.** F. Kreuzer (Dept. Physiol., Dartmouth Med. Sch., Hanover, N.H., U.S.A.). *Experientia*, 1957, **13** (7), 300 (in English).—In the simple and reliable method described, a Clark's platinum electrode is used. The values obtained compare satisfactorily with those of other methods. The time needed for one sample determination and the preparation of one calibration curve is $\simeq$20 min. P. Nicholls

1942. **Improved complexometric determination of potassium in blood serum.** H. Flaschka and A. Holasek (Med. Chem. Inst., Univ. Graz, Austria). *Hoppe-Seyl. Z.*, 1957, **308** (2–4), 183–186.—The method previously described (*cf. Anal. Abstr.*, 1957, **4**, 604) for determining K by complexometric titration of the cobalt in the cobaltinitrite ppt. has been improved. Filtration through sintered glass has been substituted for centrifuging as a more rapid means of separating the ppt., and murexide has been replaced as an indicator by 1-(2-pyridylazo)-2-naphthol, which gives a better end-point but cannot be used for the direct titration of cobalt. After the cobaltinitrite ppt. has been dissolved, the indicator is added, together with the copper complex of EDTA. The copper is displaced from the complex by the cobalt to form a copper complex with the indicator, which can be titrated with EDTA. The end-point is marked by a colour change from violet to yellow. F. Powell

1943. **Use of diphenylcarbazone for the photometric micro-determination of copper in blood, urine and tissue.** L. N. Lapin (Biochem. Lab., Samarkand Med. Inst.). *Biokhimiya*, 1957, **22** (5), 825–829.—Diphenylcarbazone (**I**) forms benzene-soluble complexes with Cu^{2+} at pH 4 to 5. The reaction is highly specific and sensitive, no interference being caused by Ag^{+}, Zn^{2+}, Al^{3+}, Sn^{2+}, Bi^{3+}, Mn^{2+}, Fe^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cr^{3+}, Ti^{4+}, $AuCl_4^-$, VO_3^-, $SbCl_4^-$, $Cr_2O_7^{2-}$, WO_4^{2-}, F^-, $PtCl_6^{2-}$ or $B_4O_7^{2-}$ under the conditions indicated. Molybdenum, Cd and Pb form colours with **I** only in high concn. (1000 times the concn. in biological material). The copper complex in benzene obeys Beer's law. The extinction of the benzene soln. is measured at 540 mμ. The error of the determination is $\gtrsim \pm 3\%$. C. D. Kopkin

1944. **Application of the entrainment of arsenic by nascent sulphur to its determination in several liquids and biological products.** V. Brustier and G. Pitet (Lab. de Chim. Anal. de Toxic. et Hyg. Ind., Fac. de Méd. et de Pharm., Toulouse, France). *Ann. Pharm. Franç.*, 1957, **15** (7–8), 457–461.—Arsenous sulphide is co-pptd. with S by the successive and simultaneous passage of Cl and H_2S through an aq. soln. The ppt. is centrifuged and introduced into the crater of a graphite electrode of an arc (Pfeilsticker intermittent arc, Hilger spectrograph). This entrainment with S increases the sensitivity of the spectrography more than entrainment with $Fe(OH)_2$, $Fe(OH)_3$ or Hg_2Cl_2. *Procedure for inorganic liquids*—To the sample (*e.g.*, H_2O, H_2O_2 or HNO_3) and standards containing 0 to 6 μg of As are added 5 μg of Sn and 2·5 μg of Au in soln., as internal standards, and the vol. is made up to 20 ml. Through each tube is bubbled H_2S in the cold for 10 min. and then a mixture of Cl and excess of H_2S (both As-free) for 30 min. Finally H_2S is passed alone again for 10 min. This procedure is necessary for the complete reduction and pptn. of arsenates. The ppt. is centrifuged and after spectrography the micro-densitometric measurements of the plates are compared; the lines used are As 2349·84 A, Sn 2334·81 A and Au 2352·65 A. *For urine*—A stream of Cl is used first to destroy urea, and octyl alcohol is added to prevent frothing; H_2S is then passed in the cold for 15 min. to prevent loss of volatile arsenic halide, and the ppt. is formed and treated as before. *For blood*—Blood (1 ml) is diluted to 20 ml and decolorised by passing Cl, and then H_2S for 15 min. in the cold, and the ppt. is formed and treated as before. The sensitivity limits are 0·5 μg per 25 ml for urine and 1 μg per ml for blood. E. J. H. Birch

1945. **Determination of pyrazinamide. II. Determination of pyrazinamide in urine and blood.** Kiichiro Kakemi, Toyozo Uno and Hitoshi Sezaki (Univ. Kyoto, Japan). *J. Pharm. Soc. Japan*, 1956, **76**, 906–909.—Separate determinations of pyrazinamide (**I**) and its *in vitro* decomposition product, pyrazinoic acid (**II**), in the urine and blood appearing after the oral administration of **I**, were carried out by means of the bright reddish-orange coloration formed by the reaction of **I** and **II** with sodium nitroprusside in Na_2CO_3 soln. The urine sample or the citrated haemolysed blood sample, which has been deproteinised with methanol, is passed through a column of Amberlite IRA-400, and **I** is determined in the eluate. The **II** adsorbed on the resin is eluted with warm NaCl soln. and determined in the eluate. It was found that the blood level of **I** is far higher than that of **II** at any time; **II** was rarely detected. In contrast, the level of **II** in the urine is far higher than that of **I**. Chem. Abstr.

1946. **Test for the determination of taurine in urine.** E. I. Pentz, C. H. Davenport, W. Glover and D. D. Smith (Argonne Cancer Res. Hosp., Chicago, Ill., U.S.A.). *J. Biol. Chem.*, 1957, **228** (1), 433–445.—The test and the determination depend on the fact that taurine is not adsorbed on the acid form of Dowex 50 and that the coloured derivative formed by reaction with 1-fluoro-2:4-dinitrobenzene is determined spectrophotometrically at 355 mμ. Cysteic acid also gives a colour under the same conditions, but the test is reasonably specific for taurine because cysteic acid is normally present in only small amounts in urine. J. N. Ashley

1947. **A simplified micro-method for the colorimetric determination of total acetone bodies in blood.** N. Bakker and R. R. White (Animal Research Station, Agric. Dept., Hamilton, N. Zealand). *N.Z. J. Sci. Tech., B*, 1957, **38** (9), 1001–1008.—The method described, in which only 1 ml of blood is used, is suitable for handling a large number of samples at a time. Acetoacetic acid and β-hydroxybutyric acid are converted into acetone by heating the sample in an autoclave, first with 11·7 *N* H_2SO_4 and then with acid dichromate. The acetone formed is coupled with salicylaldehyde in alkaline soln., and the colour is read without solvent extraction. The recovery of acetone and acetoacetic acid is complete, and of β-hydroxybutyric acid is 93%. I. Jones

1948. **Micro-determination of acetone and acetoacetic acid in blood by means of a modification of the acetoacetic acid determination of F. Bahner.** M. Böhm and H. Zimmermann (Max Planck Inst. für Ehrnährungsphysiologie, Dortmund). *Hoppe-Seyl. Z.*, 1957, **308** (2–4), 132–135.—The method of Bahner (*Biochem. Z.*, 1957, **323**, 318), by which acetoacetic acid is determined by conversion into acetone and collection of the acetone in alkaline salicylaldehyde, has been modified by an alteration in the construction of the special apparatus so as to permit the determination of acetone itself. The technique requires only 0·1 ml of blood and the error is ±2%. F. Powell

1949. **Creatine and creatine phosphate in normal and protein-depleted rats. [Determination of creatine phosphate.]** J. F. Van Pilsum (Dept. of Physiol. Chem., Med. Sch., Univ. of Minnesota, Minneapolis, U.S.A.). *J. Biol. Chem.*, 1957, **228** (1), 145–148.—The tissue is extracted with cold 0·6 *N* $HClO_4$ and the extract is neutralised to a pH of 7 to 8. This extract contains creatine phosphate plus creatine. The diluted extract is run down a column of Dowex 50 which adsorbs the creatine, and the eluate contains the creatine phosphate which is then determined by a standard method. J. N. Ashley

1950. **Determination of adenosine polyphosphates in mouse brain.** F. Leusher and C. J. Estler (Pharm. Inst. der Univ., Erlangen). *Hoppe-Seyl. Z.*, 1957, **308** (2–4), 141–146.—The enzymic acetylation of co-enzyme A is dependent on adenosine triphosphate (**I**). If the product is allowed to react with choline in the presence of choline acetylase, then acetylcholine will be produced. The rate of production of acetylcholine can be used as a measure of the concn. of **I**. By biological means, 0·02 μg of acetylcholine can be determined with an error of ±0·002 μg. The specificity of the method has been proved by paper-chromatographic separation of the **I** in a di*iso*propyl ether - formic acid system and determination of the activity of the portion containing the **I**. Adenosine diphosphate also reacts and can be determined after chromatographic separation from **I** in acetone - 15% aq. trichloroacetic acid (65:35, v/v). F. Powell

1951. **Determination of spermine, spermidine and other biological amines after paper-electrophoretic separation, and their proportions in animal organs.** F. G. Fischer and H. Bohn (Chem. Inst. der Univ., Würzburg). *Hoppe-Seyl. Z.*, 1957, **308** (2–4), 108–115.—Paper electrophoresis at 50 mA for 2 hr. in various pyridine - citric acid buffer mixtures, the composition of which is given, was found to be a satisfactory method of separating the spermine bases. Bovine pancreas, liver and lung, porcine pancreas and brewer's yeast were examined by this technique and a table is presented of the results, which showed some differences from those obtained by the use of other techniques. The authors consider the electrophoretic figures to be more reliable. Details are given of the procedures for the preliminary extraction of the various tissues and of the electrophoretic separation. The bases were located by their reaction with ninhydrin, and quant. determination was carried out by converting the ninhydrin derivative into the red copper complex, which was eluted with methanol, and its extinction measured at 506 mμ. The error is ±5%. F. Powell

1952. **Quantitative determination of amino acids on completely ninhydrin-developed chromatograms.** E. A. Ermakova (Lab. of Food Hygiene, Uzbek Research Sanitary Inst., Tashkent). *Biokhimiya*, 1957, **22** (5), 917–923.—The reaction of ninhydrin with amino acids on paper is studied. It is established that the optimum concn. of ninhydrin is 0·5 to 0·6% in butanol saturated with water, that the colour of the ninhydrin-developed chromatogram is most intense 72 hr. after the end of the 18-hr. development period, and that the colour of the water extract of the developed spot is stable for 5 hr., which is sufficient for analysis. C. D. Kopkin

1953. **Fundamental studies in paper chromatography. III. The effect of temperature on the separation of amino acids.** Shiro Harasawa and Keiji Danjo (Fac. of Engng, Yamaguchi Univ., Ube, Yamaguchi Prefecture). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (8), 1103–1106.—Chromatographic separation of amino acids was examined at 5° to 40° with three developers: (*i*) butanol - acetic acid - water (4:1:1, by vol.); (*ii*) phenol - aq. NH_3 soln. (0·1%) (4:1); (*iii*) pyridine - aniline - water (9:1:2). By the use of (*i*), little change in R_F was observed with temp. The R_F value obtained with (*ii*) increases slightly with increasing temp., but this does not have any significant influence on the chromatographic separation, except for glutamic acid at >20°. By the use of (*iii*), an overall increase in R_F is observed with rise of temp.; two max. points appear at 18° and 32° on the R_F *vs.* temp. diagram for each amino acid.

IV. The effect of changes in the constitution of solvent on the separation of amino acids. Shiro Harasawa and Keiji Danjo. *Ibid.*, 1957, **78** (8), 1106–1108.—By the use of (*i*), the R_F value first increases with increase in acetic acid [from 10 to 25 ml per 125 ml of butanol - water (4:1, by vol.)], then remains constant and again increases (>40 ml per 125 ml of the mixture). The R_F value obtained with (*ii*) increases slightly with the content of 0·1% aq. NH_3 soln. By the use of (*iii*), the R_F value increases with water content and decreases with increase in aniline. The extent to which such a change takes place is almost the same for the different amino acids. K. Saito

1954. **A method for the determination of peptides in presence of free amino acids.** A. Markovitz and D. Steinberg (Lab. of Cell. Physiol., Nat. Heart Inst., Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1957, **228** (1), 285–293.—A method is described which will permit determination of small amounts of peptides or other bound amino acids in the presence of a large excess of free amino acids. It is based on the preliminary destruction of the free amino acids by reaction with ninhydrin under conditions that favour only minimum losses of any peptide-bound amino acids. After removal or destruction of the excess of ninhydrin and subsequent acid hydrolysis of the peptides, a second reaction is carried out with ninhydrin, and the intensity of the coloured product is determined colorimetrically. J. N. Ashley

1955. **Quantitative determination of C-reactive protein [in serum].** J. Zach (Med. Poliklinik, Univ. Würzburg, Germany). *Klin. Wochschr.*, 1957, **35** (18), 936–937.—The method of Anderson and McCarty (*Amer. J. Med.*, 1950, **8**, 445) is modified for greater accuracy. A serial dilution of the serum (1:10, 1:20 . . . 1:640) is made in normal

saline; 30 μl of each dilution is mixed with an equal vol. of specific antiserum in a capillary and allowed to stand for 24 hr. The titre is then read as the lowest dilution showing no ppt. or turbidity.
H. F. W. KIRKPATRICK

1956. **Subfractionation and improved resolution of serum proteins by means of paper electrophoresis.** W. Wurm and R. Straus (St. Joseph Hosp., Burbank, Calif., U.S.A.). *Amer. J. Clin. Path.*, 1957, **27**, 691–700.—Electrophoresis, for 9 hr. at 10 mA and 470 V on paper maintained horizontally even when drying, enabled human serum to be easily fractionated into as many as 8 protein fractions, *i.e.*, albumin and α_1-, α_2-(2), β_1-, β_2-, γ_1- and γ_2-globulins. The use of special buffers and other refinements was not necessary. (For apparatus see *Anal. Abstr.*, 1957, **4**, 1939.) NUTR. ABSTR.

1957. **Ultra-micro method for the determination of total cholesterol in bile, based on the Tschugaeff colour reaction.** K. H. Jørgensen and H. Dam (Polytech. Inst., Copenhagen, Denmark). *Acta Chem. Scand.*, 1957, **11** (7), 1201–1208.—The Tschugaeff colour reaction is adapted to the determination of cholesterol in very small quantities of bile such as that obtained from hamster gall-bladders. *Procedure*—The bile (0·003 to 0·010 ml) is introduced into a centrifuge tube (about 7 ml capacity) and treated with 0·5 ml of 0·1 *M* KOH. By shaking and centrifuging, the liberated cholesterol is extracted into $CHCl_3$ and washed with 0·5-ml portions of water, still in the same tube. After evaporation of the $CHCl_3$ under vacuum, the residues in the tubes (two bile, one standard and one blank) are treated with 0·1 ml of a freshly prepared mixture of $CHCl_3$ - acetyl chloride - 20% $ZnCl_2$ soln. in acetic acid (3 : 1 : 1), and heated at 65° on a water bath after sealing the tubes with a polyethylene stopper and glycerol - starch grease. The extinctions are then measured within 15 min. at 522 and 440 mμ (max. and min. of the absorption curve, respectively). Beer's law is obeyed between concn. of 2 and 32 μg of cholesterol in a total volume of 0·5 ml, but a yellow substance accompanies the bile cholesterol, which can be corrected for from a comparison of the extinctions of standard and bile cholesterol at the two wavelengths quoted. The correction factor for this extraneous colour is 0·8 to 0·9 at the peak (522 mμ). The mean deviation between individual observations of a duplicate analysis is 5·7%. E. J. H. BIRCH

1958. **Determination of 17-oxosteroids (17-ketosteroids) and 17-oxogenic steroids (17-ketogenic steroids).** A. E. Kellie (Courtauld Inst. of Biochem., Middlesex Hospital Medical School, London). *Analyst*, 1957, **82**, 722–734.—The structure of the precursors of 17-oxosteroids and 17-oxogenic steroids is discussed. Determination of the 17-oxosteroids in urine by enzymic hydrolysis of the 17-oxosteroid conjugates with β-glucuronidase and catalytic hydrolysis of the 17-oxosteroid sulphates by dioxan in the presence of trichloroacetic acid is described. Of the large number of chromatographic systems available for separation of mixed 17-oxosteroids, the most successful is gradient elution from alumina with ethanol - benzene. Methods applicable to urine are applicable to blood, but are complicated by the small amount of 17-oxosteroids present and by the presence of protein and fat. Methods of identification of individual 17-oxosteroids are discussed and finally the determination of non-ketonic steroid alcohols. A. O. JONES

1959. **Quantitative determination of corticosteroids. I. Quantitative determination of conjugated corticosteroids with use of β-glucuronidase.** Akira Kumagami, Masaki Otomo, Saburo Yano, Nozomu Tskeuchi and Koichiro Yagura (Osaka Univ. Med. School, Japan). *Folia Endocrin. Jap.*, 1956, **32**, 538–547.—When the free form of corticosteroids (**I**) is determined by Miyake's modification of Daughaday's formaldehydogenic method (Daughaday and MacBryde, *J. Clin. Invest.*, 1950, **29**, 591), the calf spleen β-glucuronidase preparation (**II**) (*cf.* Fishman, *J. Biol. Chem.*, 1939, **127**, 367) shows the optimum pH at 5·0 in hydrolysis of the glucuronide of **I** in urine. For 10 ml of normal urine 4000 units of **II** is required. For the quant. hydrolysis of glucuronides of **I**, the action of **II** is improved to proceed over a $CHCl_3$ layer in a separating funnel by occasional shakings for 40 hr. The average values of 7 urines were—free 0·91 ± 0·21, conjugated 7·10 ± 1·78, total 8·01 ± 1·89 mg per day. Cortisone acetate (50 to 100 mg) and adrenocorticotrophin (80 units) injections increase **I**, especially in its free form. For the estimation of **I** in blood, the following improved procedures have been devised. Evaporate the solvent of the Bloor filtrate, dissolve the residue in water, extract the free **I** with $CHCl_3$ and follow by $MgCl_2$ treatment in acetone, benzene extraction, water re-extraction, and formaldehydogenic reaction, successively. For estimation of the conjugated **I**, add **II** to the remaining aq. layer after $CHCl_3$ extraction of the free **I**, extract the hydrolysed free **I** with $CHCl_3$, extract with benzene, re-extract with water, and run the formaldehydogenic reaction. The average values of 10 blood samples were—free 0·37 ± 0·14, conjugated 0·59 ± 0·29, total 0·96 ± 0·32 mg per 100 ml. CHEM. ABSTR.

1960. **Purification and properties of pancreatic cholesterol esterase. [Determination of enzymic activity.]** H. H. Hernandez and I. L. Chaikoff (Dept. of Physiol., Univ. of California Med. Sch., Berkeley, U.S.A.). *J. Biol. Chem.*, 1957, **228** (1), 447–457.—A rapid and simple turbidimetric method is described for the assay of cholesterol esterase. It is based on the fact that a mixture of cholesterol and an emulsifying agent stabilises emulsions of oil in water. Replacement of the cholesterol by its acetate or stearate fails to give a stable emulsion. A water-clear emulsion is prepared from cholesterol and oleic acid in phosphate buffer containing Na taurocholate. When cholesterol esterase is added to this emulsion a turbidity develops, and this is measured in a Klett colorimeter. The turbidity is proportional to the amount of enzyme added.
J. N. ASHLEY

Drugs

1961. **Quantitative determination of alkaloids from paper chromatograms by a photographic method.** R. Miram and S. Pfeifer (Pharm. Inst., Humboldt Univ., Berlin). *Pharm. Zentralh.*, 1957, **96** (8–9), 457–462.—A method is described for the determination of alkaloids, *e.g.*, papaverine and codeine, by means of a modified Dragendorff reaction, followed by the preparation of contact negatives on orthochromatic film. The alkaloid (5 to 50 μg) gives grey spots on a black background, the intensity of which is measured. D. P. FELIX

1962. **Quantitative determination of codeine and dihydrocodeine in antitussive and expectorant drugs by paper chromatography.** Haruyo Asahina

and Masako Ono. *Eisei Shikenjo Hókoku*, **1956**, (74), 61–64.—Paper chromatography is applied with a solvent comprising butyl alcohol - aq. NH_3 (28%) - H_2O (50:9:15). Spots of codeine (**I**) at R_F 0·84 and of dihydrocodeine (**II**) at R_F 0·86 are stained by potassium iodoplatinate soln., which is followed by absorptiometric measurement at 283 mμ for **I** and 282 mμ for **II**. The Beer - Lambert law applies to **I** and **II** from 10 to 60 μg.

CHEM. ABSTR.

1963. **Extraction, chromatographic separation and quantitative colorimetric determination of galegine.** M. Tiberio (Ist. Sper. Zootec., Rome). *Ann. Sper. Agr.*, **1957**, **11**, 1045–1054.—The alkaloid galegine is supposed to be responsible for the stimulation of milk production by *Galega officinalis*. Galegine has been extracted from *Galega officinalis* L., in which it occurs in almost equal amounts in seeds, leaves and flowers. Almost pure galegine can be obtained by chromatography on a column of cellulose with a mixture of butanol, acetic acid and water. By the Sakaguchi reaction, characteristic of mono- and di-substituted guanidine bases, galegine was estimated colorimetrically by comparison with soln. of arginine hydrochloride. NUTR. ABSTR.

1964. **Reactions with xanthhydrol. I. Photometric determination of reserpine.** Z. Jung (State Inst. for Control of Drugs, Prague). *Československ. Farm.*, **1957**, **6** (6), 299–302.—The indole component of reserpine (**I**) reacts with xanthhydrol (**II**) to form a coloured compound. Protoveratrine, methyl phenidate, dihydrallazine and hexamethonium do not interfere. **I** is extracted from coloured tablets with a satd. soln. of $NaHCO_3$ into $CHCl_3$. Extremely small amounts of **I** are extracted from the powdered tablets into a 25% (w/v) soln. of tartaric acid and then into $CHCl_3$. Coloured components are removed by shaking the $CHCl_3$ extract with a soln. of $NaHCO_3$. *Procedure*—**I** (50 to 250 μg) is dissolved in 5 ml of the freshly prepared reagent [40 mg of **II** dissolved in 100 ml of glacial acetic acid and 1 ml of HCl (35% w/v)]. The test-tube is stoppered and heated for 15 min. in a boiling-water bath. The extinction of the soln. (cooled in an ice bath) is measured at 515 mμ against a blank, and the concn. is calculated from a calibration curve.

II. Photometric determination of yohimbine. Z. Jung. *Ibid.*, **1957**, **6** (8), 436–437.—The indole component of yohimbine (**III**) reacts with **II** with formation of a coloured compound. *Procedure*—**III** (50 to 150 μg) is heated in a test-tube with 5 ml of a freshly prepared soln. of **II** (0·4%, in glacial acetic acid containing 1% (v/v) of HCl] in boiling water for 15 min. The soln. is cooled and the extinction is measured at 515 mμ. The colour is stable for 5 hr. The procedure for the analysis of tablets and injections is given in detail. (*Cf.* Part I.)

J. VOLKE

1965. **Determination of reserpine, ajmaline and serpentine in mixtures.** F. Machovičová, V. Parrák, O. Lišková and J. Ružičková (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Československ. Farm.*, **1957**, **6** (10), 584–586.—With formamide as the stationary phase these alkaloids were separated by paper chromatography. Chloroform satd. with formamide and descending chromatography on Whatman No. 1 paper gave the following R_F values—reserpine, 0·90, ajmaline, 0·52, and serpentine, 0·04. The eluted compounds were caused to react with methyl orange to form an addition compound and determined colorimetrically. The optimal pH value is between 4 and 6, and small amounts (50 to 100 μg) of alkaloids may be determined.

J. VOLKE

1966. **The paper-chromatographic separation of methadone hydrochloride, ephedrine hydrochloride and hyoscine hydrobromide.** F. Abaffy and S. Kveder (Anal. Lab., "Pliva," Zagreb, Yugoslavia). *Acta Pharm. Jugoslav.*, **1956**, **6**, 207–209.—Circular, descending and ascending techniques were used on Whatman No. 1 filter-paper, with butyl alcohol - glacial acetic acid - H_2O (10:1:4, by vol.) as solvent. A mixture of butyl alcohol, formic acid and H_2O could also be used. The R_F values were determined at 20° ± 2°. For the detection of ephedrine hydrochloride, ninhydrin was used, whereas for the identification of methadone hydrochloride and hyoscine hydrobromide, a modified Dragendorff reagent was useful.

CHEM. ABSTR.

1967. **Colorimetric determination of rutin.** J. Davídek and J. Fragner (Res. Inst. Food Technol., Prague). *Československ. Farm.*, **1957**, **6** (8), 449–450.—Rutin reacts with diazotised *p*-aminobenzoic acid with formation of a coloured product. *Procedure*—*p*-Aminobenzoic acid (0·5%) (1 ml) in H_2SO_4 (10%) is measured into a 25-ml flask, $NaNO_2$ (0·2%, 2 ml) is added, and the soln. is agitated until the light-yellow colour is discharged. The methanolic soln. of rutin is added, and the sample is made alkaline with NaOH (10%, 5 ml) and diluted to 25 ml. The extinction is measured at 420 mμ.

J. VOLKE

1968. **Paper-chromatographic identification of polypeptidic Gram-positive-inhibiting antibiotics.** N. Snell, K. Ijichi and J. C. Lewis (U.S. Dept. Agric. Res. Serv., Albany, Calif.). *Appl. Microbiol.*, **1956**, **4**, 13–17.—The method is based on the differentiation of the antibiotics by ascending paper chromatography, with *tert.*- or *n*-butanol, methanol or acetone containing 2 to 10% of acetic acid as solvent. A few ml of aq. NH_3 is placed in the cell to maintain an atmosphere containing NH_3. The papers are dried, with intermittent exposure to steam to remove acetic acid, and then placed on an agar nutrient medium inoculated with a suitable test organism, *e.g.*, *Bacillus megatherium*, to locate the antibiotic. A key for identifying the antibiotics and a table of R_F values are presented.

A. G. POLLARD

1969. **Quantitative determination of zinc [in protamine zinc insulin] by paper chromatography.** Seiichi Okazaki. *Eisei Shikenjo Hókoku*, **1956**, (74), 51–56.—The content of Zn in commercial protamine zinc insulin (**I**), containing 20 units per ml, is determined as follows. Add dil. HCl (in 0·01-ml portions) to 5 ml of **I** to give a clear soln., place 25 and 50 μl on a strip (1 cm × 40 cm) of filter-paper (Toyo Roshi No. 50) 6 cm from the end. Develop upwards with butyl alcohol - water - 12 *N* HCl (10:3:2) for 14 hr., dry, immerse in 0·05% dithizone in $CHCl_3$ for 10 sec., dry and determine the area of purple spots or estimate the intensity of reflection of the spot by 530 mμ monochrome, taking non-coloured filter-paper as a control. Repeat this procedure with 1 to 16 μg of Zn as standard soln. The area and reflection intensity demonstrate a linear relation to logarithmic concn. of Zn. The content of Zn in **I** is determined as 0·428 and 0·407

mg per 100 units of **I** by the area-measuring and reflection methods, respectively. These values are approx. the same as those obtained by the methods of the U.S. Pharmacopeia, International Pharmacopeia or Japanese National Formulary II.

CHEM. ABSTR.

1970. **Analysis of Schleich's anaesthetic mixture by gas - liquid partition chromatography.** L. Domange and S. Longuevalle (Lab. Nat. de Contrôle des Médicaments, Paris). *Ann. Pharm. Franç.*, 1957, **15** (7–8), 448–454.—The components (nominally diethyl ether 66·66%, $CHCl_3$ 22·22%, chloroethane 11·11%) of Schleich's mixture are determined with an accuracy of $\simeq \pm 5\%$ by gas chromatography, with a silicone (E.301) on celite as the liquid phase, and N at 0·6 litre per min. at 36° with a pressure drop of 325 to 100 mm. The retention times for each component are characteristic and under these conditions were 3 min. for chloroethane, 5·5 min. for diethyl ether and 12 min. for $CHCl_3$. The mixture for analysis (0·02 ml) is injected from a syringe kept at 0°. The apparatus is calibrated in terms of the ratios of the areas of the peaks by the use of known mixtures. The use of added $CHCl_3$ as an internal standard appears to be without advantage.

E. J. H. BIRCH

1971. **Paper-chromatographic determination of the decomposition product of procaine hydrochloride in its solution.** Ju Fukuzawa and Toshi Fujiyo (Chiba Univ. Hosp., Japan). *Yakuzaigaku*, 1956, **16**, 22–24.—Procaine hydrochloride decomposes into *p*-aminobenzoic acid (**I**) and diethylaminoethanol. The test soln. is developed on filter-paper with water - ethanol (3:1) and sprayed with dil. HCl (10 ml), 10% $NaNO_2$ soln. (3 drops), 25% urea soln. and 0·2% Tsuda's reagent successively to colour the decomposed **I**. This colour is measured in a Dubosq colorimeter. By this method it was found that in the heat sterilisation of procaine hydrochloride soln. at 100° for 30 min., approx. 1 to 2% was decomposed, and this decomposition was accelerated in acid medium. CHEM. ABSTR.

1972. **The analytical behaviour of Wofacain A [diethylaminoethyl 4-butylaminosalicylate hydrochloride].** M. Hädicke (Staat. Inst. Jena, Germany). *Pharm. Zentralh.*, 1957, **96** (8–9), 396–401.—Qual. and quant. procedures are given for the physical and chemical analysis of Wofacain A and methods of distinguishing it from procaine and chlorprocaine are established. D. P. FELIX

1973. **Proposals for the chemical investigation of some sympathomimetic drugs of the German Pharmacopoeia.** W. Awe and H. Stohlmann (Techn. Hochsch., Braunschweig, Germany). *Pharm. Zentralh.*, 1957, **96** (8–9), 371–381.—Bromatometric methods have been applied to the determination of some sympathomimetic drugs, *e.g.*, phenylephrine and derivatives and pholedrine. D. P. FELIX

1974. **Determination of pyrazinamide. III. Simultaneous determination of pyrazinamide and isoniazid.** Hitoshi Sezaki and Shozo Sakata (Kyoto Univ., Japan). *Yakuzaigaku*, 1957, **16** (4), 26–28.—The simultaneous determination of each component in a mixed preparation of pyrazinamide (**I**) and isoniazid (**II**) was carried out. **II** was first determined by the colour produced with Na 1:2-naphthaquinone-4-sulphonate in alkaline medium, the extinction being measured at 480 mμ, then **II** was decomposed with bromine water, and the soln. was treated with Amberlite IRA-400. The eluate was used for the determination of **I** by the coloration given with sodium nitroprusside, the extinction being measured at 494 mμ. *iso*Nicotinic acid and pyrazinoic acid in the sample do not interfere.

CHEM. ABSTR.

1975. **Separate determination of each component from a mixture of pyrazinamide and isoniazid. I. Determination of pyrazinamide.** Hiroshi Sakurai, Takeshi Kimura and Setsuya Senoo (Sankyo Shinagawa Factory, Tokyo). *Yakuzaigaku*, 1956, **16** (2), 39–41.—The maximum u.v. absorption of pyrazinamide (**I**) is at 269 mμ and 311 mμ, and of isoniazid (**II**) at 267 mμ. The mixture of **I** and **II** ($\simeq$100:15), $\simeq$100 mg, is dissolved in 0·1 *N* HCl, made up to 1 litre, and the absorbancy measured at 311 mμ. Data are given of the relation between concn. and absorbancy of **I**.

II. Determination of isoniazid. Hiroshi Sakurai and Yoshio Sahashi. *Ibid.*, 1956, **16** (3), 48–50.—**II** in a complex **I** - **II** tablet was determined by oxidation - reduction titration with 0·05 *N* $K_3Fe(CN)_6$ in 15% KOH soln. In this titration, **I** seems to undergo decomposition, but there is no effect on potential if the titration is made 10 min. later. The presence of lactose and talc does not cause interference. Starch gelatinises with 15% KOH soln., but its interference can be avoided by alkalisation after filtration. The accuracy of this method was examined with synthetic samples and the error found was $< \pm 0{\cdot}7\%$. CHEM. ABSTR.

1976. **Determination of 2-mercapto-1-methyliminazole (Metothyrin).** J. Blaček, J. Kráčmar and Z. Stejskal (State Inst. for Control of Drugs, Prague). *Českosl. Farm.*, 1957, **6** (8), 441–442.—2-Mercapto-1-methyliminazole (**I**) is titrated with 0·1 *N* iodine in sodium bicarbonate soln. with starch as indicator or in 2 *N* NaOH with potentiometric control (a platinum and a calomel electrode). When titrating $\simeq$10 mg of **I** the accuracy is within $\pm 0{\cdot}5\%$; it is greater with larger amounts.

J. VOLKE

1977. **Quantitative determination of Rivanol (lactoacridine) by the use of iodine chloride.** P. P. Suprun. *Med. Prom. SSSR*, 1957, (2), 49–50; *Ref. Zhur., Khim.*, 1957, Abstr. No. 63,822.—Dissolve 0·05 to 0·1 g of Rivanol in a 100-ml flask with 10 to 15 ml of warm water, and add 25 ml of a 0·1 *N* soln. of ICl (shake a mixture of 3·5 g of KIO_3, 5·5 g of KI and 40 ml of 37% HCl until a clear soln. is obtained, add 15 ml of $CHCl_3$ and a 1% soln. of KIO_3 till the $CHCl_3$ layer is colourless; remove the aq. layer and make up to a litre). Add 50 or 60 ml of hot water to the mixture and shake. After 15 or 20 min., cool, make up to volume and filter, discarding the first portions of filtrate. To 50 ml of filtrate add 10% KI soln. (10 ml) and titrate with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. The method is rapid and accurate.

C. D. KOPKIN

1978. **Separation of sulphonamides by chromatography and electrophoresis on paper.** M. Cormier, P. Jouan and G. Dancoisne (Fac. de Méd. et de Pharm., Rennes, France). *Ann. Pharm. Franç.*, 1957, **15** (3), 176–183.—Paper chromatography, with *n*-butanol or *tert.*-amyl alcohol saturated with 2 *N* aq. NH_3 as solvent, effects the separation of sulphanilamide (**I**), sulphaguanidine (**II**), sulphamethiazole (**III**), sulphathiazole (**IV**) and sulphapyridine (**V**). Sulphasolucin (**VI**), N-3:4-dimethylbenzoylsulphanilamide (**VII**) and **I** give comparable

R_F values in the amyl alcohol solvent. Electrophoresis is carried out in 0·02 *M* aq. ammonium carbonate or 0·012 *M* NaOH, at 700 V and 7 mA for 4 hr. and 1 hr., respectively. In this way, sulphadimerazine, **IV** and **V** may be separated, and **I**, **II** or **VI** may be separated from **III**, **IV**, **V** or **VII**. The spots are detected by modified Ehrlich reagent or a nitrite - 2-naphthol diazo reagent.
E. J. H. Birch

1979. **Complexometric determination of bismuth. Applications to the control of medicaments.** J. Delga and J. Storck (Lab. de Chim., Val-de-Grâce, France). *Ann. Pharm. Franç.*, 1957, **15** (5), 299–303.—*Procedure*—The sample (≡ 30 to 80 mg of Bi) is dissolved in 2 ml of 10 *N* HNO_3, and 40 ml of a glycine buffers soln. containing NaCl and thiourea, and 5 ml of 2·9 *N* aq. NH_3 are added. The soln. is then titrated with 0·05 *M* EDTA (disodium salt) until the yellow colour of the Bi - thiourea complex is discharged. When the Bi is contained in org. material which may interfere either with the colour or the buffering, the org. matter is destroyed with gentle ignition, and the residue is taken up with HNO_3, buffered and titrated as before. When the method was applied to several synthetic mixtures, there was no interference from certain drugs and Ca, Mg, Al and kaolin. E. J. H. Birch

1980. **Determination of 7-oxocholesterol in wool wax.** A. H. Milburn and E. V. Truter (Textile Chem. Lab., Univ., Leeds, England). *J. Appl. Chem.*, 1957, **7** (9), 491–497.—Digitonin forms an insoluble complex with 7-oxocholesterol which shows the same absorption max. at 240 mμ as 7-oxocholesterol. Free 7-oxocholesterol in a mixture of sterols dissolved in 90 to 95% aq. ethanol can be pptd. with a 1% soln. of digitonin in 90 to 95% aq. ethanol. A correction of 16 mg per 100 ml of ethanol must be made for the solubility of the digitonide. Alternatively, sufficient water to cause a faint turbidity can be added after the digitonin, and the soln. evaporated until a moist residue is obtained. This is washed with ether followed by hot water. The final residue is the digitonide. An aliquot of the weighed digitonide is dissolved in a known vol. of ethanol and the absorption is measured over the range 230 to 250 mμ. Esters of 7-oxocholesterol can be hydrolysed in ether soln. with a cold methanolic soln. of Na methoxide. The amount of 7-oxocholesterol in wool waxes varies between 0·7% and 2·8% with an additional 0·2% to 3·9% as esters. Not more than trace amounts occur in human sebum. The error of the determinations is ⩾ 2%. Cholestenone, which also shows a max. at 240 mμ, occurs in only trace amounts in wool wax. At least part of the 7-oxocholesterol originates by autoxidation of cholesterol, suggesting that 7-oxocholesterol is not a physiological product. G. Burger

1981. **Titrations with quinol and similar reducing agents. V. Determination of cerium in pharmaceutical preparations.** E. Krejzová, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Českosl. Farm.*, 1957, **6** (8), 438–440.—Cerous salt is oxidised to ceric salt by the action of persulphate in acidic soln. Silver ions act as a catalyst. The ceric salt is titrated with 0·1 *N* quinol, with ferroin as indicator. By this titration the content of Ce is determined, without the other rare earths. Some of the pharmaceutical preparations are decomposed by combustion or mineralised before the determination. J. Volke

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See also Abstracts—**1792**, Determination of mercury in pharmaceuticals. **1945**, Determination of pyrazinamide in urine and blood.

Food

1982. **A rapid method of estimation of ionisable ash in molasses by ion exchange.** S. Mukherjee. *Sugar J.*, 1957–58, **20** (6), 10–11.—Dilute soln. of molasses, obtained from various sulphitation and carbonation factories, were passed through a column of strong cation-exchange resin (H form) and the resulting acidity was determined by titration, after washing the resin with water. A general equation $Y = 0{\cdot}09072x - 1{\cdot}35$, where Y is the ash (%) and x is the vol. (ml) of N acid released from the resin per 100 g of sugar, was fitted by statistical analysis of the tabulated results. The differences between the calculated ash content and the ash content found by this method ranged from −0·61 to +0·99. Sugar Ind. Abstr.

1983. **Electrophoretic separation of amino acids in sugar-factory products, and the identification of γ-aminobutyric acid and of ornithine.** P. Pavlas and O. Melounová-Häuslerová. *Listy Cukr.*, 1957, **73** (6), 131–135.—The electrophoretic separation was carried out at pH 5·6; neutral amino acids were then separated by paper chromatography combined with electrophoresis. In the group of lower mobility the following compounds were determined: serine, glycine, threonine, alanine, tyrosine, methionine, valine, tryptophan, phenylalanine, leucine, *iso*leucine, asparagine and glutamine; in the second group only γ-aminobutyric acid was detected. The isoelectric point of γ-aminobutyric acid was calculated on the basis of dissociation constants and its value (7·33) determined. By carrying out the electrophoresis of basic amino acids, lysine, histidine, arginine and ornithine were detected.
J. Zýka

1984. **New aspects of the evaluation of biscuit flour.** G. Clewlow and W. Dixon (Wallace and Tiernan, Ltd., Power Rd., London). *J. Sci. Food Agric.*, 1957, **8** (9), 541–547.—Two new tests are described and the apparatus is illustrated. In the "specific recovery" test a sphere of dough (15 g) prepared in a specified manner is pressed to a thickness of 35 mm and dried at 105°. The difference (in mm) between the diam. of the dry disc and 68 mm, the max. diam. of the disc of dough under load, is the specific recovery. In the "water tolerance" test, 30-g spheres of dough, of water contents differing by 1 or 2%, are pressed for 5 min. under a total load of 700 g, and the diam. of the discs are measured with the dough still under load, water contents being chosen so that the diam. ranges from 50 to 70 mm. The graph of diam. against water content is a straight line, and the slope of this, multiplied by 100, gives the water tolerance. Replication in both tests is satisfactory. A biscuit flour should have specific recovery < 18 and water tolerance ⩾ 50. The tests can be used to predict the behaviour of a flour in use, and for the determination of the SO_2 treatment required to produce a biscuit flour of given characteristics.
R. E. Essery

1985. **Quantitative determination of DDT in milled cereals and baked products and its retention in bread.** G. Bruckner, R. Flatow and M. Rohrlich. *Getreide*, 1957, **7** (9), 73–77.—The methods of Schechter *et al.* and Amsden and Wallbridge for the determination of DDT were modified to permit of their use with cereals and milled products. In addition, a rapid photometric method for determining DDT was developed, based on the cloudiness produced when water was added to a DDT soln. The DDT content of flour varied between 2 and 18 mg per kg. From bread made with flour containing DDT, only about 10% of the DDT was recoverable. It was believed that the remaining 90% had not been destroyed or volatilised, but was bound in some way, which prevented its extraction by the usual method.

BRIT. BAKING IND. RES. ASS. ABSTR.

1986. **Viscometric method for the determination of α-amylase activity in individual cereal grains.** B. Belderok. *Getreide*, 1957, **7** (9), 77–80.—A viscometer, comprising a capillary tube enclosed in a thermostatically controlled jacket, and its method of use are described in detail. The quantity of starch paste required by this instrument was so small that it was possible to carry out determinations on individual grains of wheat. The examination of 50 individual grains showed that, while 92% were sound, the remainder had a higher α-amylase activity, so that bread made with flour milled from this wheat had a moist and sticky crumb. In another instance, over 50% of the grains showed a high α-amylase activity and the resulting bread was typical of that produced by flour from sprouted wheat. None of the grains examined showed any visible signs of sprouting.

BRIT. BAKING IND. RES. ASS. ABSTR.

1987. **Formol titration and its use for determination of protein in milk from individual cows.** P. Solberg, G. Syrrist and K. Steinsholt. *Meld. Norg. LandbrHogsk.*, 1957, **36** (6), 14 pp.—Protein was estimated by formol titration in 2304 samples of milk taken at regular intervals during one complete lactation from each of 308 cows and the method was compared with others. Results agreed more closely with those obtained by the Kjeldahl method than with those by the Schlossman casein method; addition of oxalate for formol titration was of no advantage. Protein was found to be calculated better from the formol titration by a regression equation than by a conversion factor. Results from individual cows varied considerably from those obtained by the Kjeldahl method. Protein calculated from formol titre rose during lactation. NUTR. ABSTR.

1988. **Determination of soluble whey protein by direct nesslerisation of Kjeldahl digests.** W. K. Stone and A. H. Holmes (Virginia Agric. Expt. Sta., Virginia Polytechnic Inst., Blacksburg, U.S.A.). *Food Res.*, 1957, **22**, 501–508.—A rapid method for the direct determination of total soluble whey protein in milk and milk products is described. *Procedure*—Determine the content of solids-not-fat and standardise a sample to contain 8·0 g of solids-not-fat and 92 g of H_2O. Mix, and warm the standardised sample to 40°; add NaCl (40 g) shaking vigorously for 40 sec.; maintain the mixture at 40° for 30 min. then filter it through a Whatman No. 2 paper into a 100-ml stoppered cylinder. Transfer an aliquot of the filtered whey into a Pyrex-glass test-tube (25 mm × 200 mm) (take 1 ml if whey is from unheated, "low" or "medium heat" non-fat dry milk, or 3 ml if from "high heat" non-fat dry milk). Prepare three blank tubes by substituting H_2O (1 ml) for the whey. Dilute each test soln. to 6 ml with H_2O, add $ZnSO_4$ soln. (5% w/v) (1 ml) and, while swirling, add 0·5 *N* NaOH (0·6 ml) (or as pre-determined, with phenolphthalein as indicator) to precipitate the proteins. Allow the tubes to stand for 5 min., rinse each with H_2O (5 ml) and centrifuge for 5 min.; draw off the supernatant liquid and re-wash the residue with H_2O (5 ml). To the centrifuged residue add H_2SO_4 (0·5 ml), and heat the tubes at an angle of 45° on a sand-bath to remove H_2O; continue the digestion for 10 min., cool and add H_2O_2 (30%) (2 drops); continue heating for 10 min., then cool the tubes to room temp. To two of the blanks add H_2O (10 ml) and standard $(NH_4)_2SO_4$ soln. to give 0·4 mg and 0·6 mg of ammonia nitrogen, respectively. Dilute the contents of all the tubes to 35 ml with NH_3-free H_2O and add Nessler's soln. (15 ml) to each. After 6 to 8 min., read the absorption at 550 mμ and determine the nitrogen content from a standard graph. H. B. HEATH

1989. **Identification and determination of formaldehyde in food products.** J. C. Godfrain, P. Bertrand and L. Liandier (École Nat. Vét., Toulouse). *Bull. Acad. Vét. France*, 1956, **29**, 521–526.—A method previously described (*Ibid.*, 1956, **29**, 367), based on the blue colour which formaldehyde gives with codeine in the presence of H_2SO_4 and an oxidising agent ($FeCl_3$), was adapted for determining formaldehyde in milk, syrups, lemonade and fruit juices. NUTR. ABSTR.

1990. **Chromatographic method for the determination and identification of preservatives in preserved foods and citrus juices.** G. Safina. *Conserve e Deriv. Agrumari*, 1956, **20**, 191–194.—Acidify 100 to 200 ml of liquid samples with a few drops of HCl or H_2SO_4 and defecate with 2 to 3 ml of Carrez's (Cu lactate) soln. to eliminate proteins. If the sample is a solid or is dense (*e.g.*, tomato paste), disperse 25 to 50 g in hot H_2O, stir for 15 min. on a water bath, filter, and acidify as above. Extract with three 25 to 50-ml portions of a mixture of equal vol. of light petroleum and diethyl ether. Filter the solvent phase, add 1 ml of 75% methanol, and allow to evaporate at room temp. Adjust the vol. to 1 ml and prepare chromatograms on Whatman No. 1 paper with pyridine - aq. NH_3 (1·5 *N*) - butyl alcohol (1:2:2) and benzene - light petroleum (40° to 70°) - methanol (2:10:1) as solvents, and with Millon's reagent, $AgNO_3$ and pyrogallol, bromocresol green, phenol red, and chloramine T as developers. *p*-Hydroxybenzoic acid and its methyl, ethyl, propyl, butyl and benzyl esters, salicylic acid, bromoacetic acid and its esters, benzoic acid, chloroacetic acid and *p*-chlorobenzoic acid were identified. CHEM. ABSTR.

1991. **Separation of permissible food dyes by two-dimensional chromatography and electrophoresis on paper.** J. R. A. Anderson, L. C. Lock and E. C. Martin (N.S.W. Univ. Technol., Broadway, Sydney, Australia). *Aust. J. Appl. Sci.*, 1957, **8** (2), 112–119.—The R_F values of 20 permissible food dyes have been determined on filter-paper by partition chromatography and their movement by electrophoresis in both acid and alkaline media has been measured. These colours have been separated from mixtures by two-dimensional

chromatography and by electrophoresis. A technique for their separation from foodstuffs by the use of butanol saturated with 2 *N* HCl as the extraction medium is described. O. M. WHITTON

1992. **Spectrophotometric method for the evaluation of vegetable-oil colours.** W. D. Pohle and S. E. Tierney (Swift & Co., Chicago, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (10), 485–489.—An improved spectrophotometric method, based on optical density at 500 mμ, is described for evaluating oil colour, amount of colour removed by bleaching, and effectiveness of bleaching earths. Data are presented on refined and bleached oils, and on possible variations in results obtained in different laboratories. S. C. JOLLY

1993. **Conductivity method for the determination of soap in refined vegetable oil.** H. Goff, jun., and F. E. Blachly (The Sharples Corp., Philadelphia, Pa., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (6), 320–323.—Published methods for the determination of soap in vegetable oil are discussed, and a simple rapid procedure based on conductivity measurement is described. Conductivity water (50 ml) is introduced into a tetraethyl-lead extractor, followed by the sample (100 g) and a further 50 ml of conductivity water. The mixture is heated under reflux for 4 min., then allowed to separate for 2 min., and 80 ml of the aqueous extract is drawn off. The resistance of the extract (at 30°) is measured by means of a conductivity bridge, and the appropriate soap content (as Na oleate) is read directly from standard curves. Variables in this method are discussed, and its adoption as an official procedure is suggested. G. HARDING

1994. **Identification of some marine-oil constituents by chromatography.** M. F. Sorrels and R. Reiser (Texas Agric. Exp. Sta., College Park, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (3), 131–134.—Vitamin-A alcohol, vitamin-A palmitate, 7-dehydrocholesterol, vitamin D_3, cholesterol, hexadecanol, cholesteryl palmitate, hexadecyl palmitate, squalene and vitamin E are separated by chromatography on glass-fibre paper impregnated with silicic acid by ascending development with either *cyclo*hexane or 1% methanol in *cyclo*hexane or 2% diethyl ether in *iso*octane. R_F values are given. The method has been applied to samples of marine oils after a preliminary separation of the components into 5 groups by chromatography on silica columns by the method of Fillerup and Mead (*Anal. Abstr.*, 1954, **1**, 1918). Identification and semi-quant. determination is possible for all the components named above except for vitamins A and D.
A. R. ROGERS

1995. **Detection of food adjuncts. I. Qualitative analysis of antioxidants by paper chromatography.** Kohei Kawata and Yutaro Hosogai. *Eisei Shikenjo Hôkoku*, 1956, (74), 239–241.—For the identification of antioxidants in foods, the following chromatographic method has been devised. Dissolve the animal or plant oils in light petroleum, extract with 72% ethanol, filter, concentrate to between 5 and 10 ml under reduced pressure at low temp., extract the residue with ethyl acetate, dehydrate, concentrate and place on a paper chromatogram. Develop with a solvent mixture of methanol - acetone - H_2O (1·0:1·0:0·8) and stain with ammoniacal $AgNO_3$ soln. Nordihydroguaiaretic acid (**I**), propyl gallate (**II**) and *iso*amyl gallate (**III**) give black spots at R_F values of 0·45, 0·66 and 0·62, respectively. Butylated hydroxyanisole (**IV**) gives a black spot at R_F 0·47 on heating at 80° to 100°. The limit of detection for **I**, **II** and **III** is 10 μg, and for **IV**, 100 μg. CHEM. ABSTR.

1996. **Determination of vitamin A by conversion to anhydrovitamin A.** P. Budowski and A. Bondi (Agric. Res. Sta., Rehovot, Israel). *Analyst*, 1957, **82**, 751–760.—Saponification of the sample and extraction of the unsaponifiable matter are carried out by standard procedures. The final extract of unsaponifiable matter is dissolved in dry benzene so that 1 ml contains between 5 and 50 i.u. of vitamin A. This soln. (1 ml) is treated with 4 ml of the catalyst (15 mg of toluene-*p*-sulphonic acid in 100 ml of dry benzene) (prep. described), and after 1 min. the catalyst is neutralised by shaking the soln. with 5 ml of 0·5 *N* NaOH. The soln. is clarified by centrifuging or settling, and the extinction of the clear liquid is measured at 399 mμ against a mixture of 1 vol. of unsaponifiable matter in benzene and 4 vol. of benzene set at 100% transmission. The method has a high degree of specificity and has been applied to various samples, including vitamin-A concentrates, fish oils, margarine, butter and poultry mashes without further purification of the unsaponifiable extracts.
A. O. JONES

1997. **Oxidation of vitamin A.** J. Čelikovský and A. Malý (Res. Inst. of Oils and Fats, Ústí nad Labem, Czechoslovakia). *Průmysl Potravin*, 1957, **8** (7), 368–371.—The method of circular (zone) chromatography on paper saturated with liquid paraffin was found to be suitable for the determination of the purity of crystallised vitamin A. The oxidation products are separated from the principal blue spot. Antimony trichloride soln. was used as the detecting agent. J. ZÝKA

1998. **A high-frequency detector for eluates from ion-exchange columns.** Tokuro Ozaki (Fac. of Educ., Shizuoka Univ., Oiwa-cho). *Japan Analyst*, 1957, **6** (6), 382–383.—A high-frequency device (*cf.* Laskowski and Putscher, *Brit. Abstr. C*, 1952, 523) for detecting colourless components was applied to the indication of elution of vitamin C and citric acid from a column of Amberlite IR-4B (50 to 60 mesh, diameter 0·9 cm, length 20 cm). Vitamin C is eluted with borate buffer of pH 5·9 until the micro-ammeter indicates its complete removal, then determined colorimetrically by the indophenol method. Citric acid is similarly eluted with *N* HNO_3 and titrated with $KMnO_4$.
K. SAITO

1999. **Determination of tocopherol in oxidised fats.** E. N. Frankel, C. D. Evans and J. C. Cowan (U.S. Dept. Agric., Peoria, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (11), 544–546.—Tocopherol (**I**) is determined by the Stern and Baxter method in which the time allowed for the Fe^{2+} - 2:2′-dipyridyl colour development is extended to 10 min. Interfering peroxides are removed by heating the oxidised oils to 210° for 10 to 15 min. *in vacuo*; recoveries of **I** are higher than by other removal methods. Heating does not regenerate **I** in such treated oils. G. R. WHALLEY

2000. **Analysis of corn oil for total tocopherols.** K. J. Hivon and F. W. Quackenbush (Purdue Univ., Lafayette, Ind., U.S.A.) *J. Amer. Oil Chem. Soc.*, 1957, **34** (6), 310–314.—In the analysis of maize oil, the usual Parker and McFarlane treatment of the oil with H_2SO_4 (*cf. Canad. J. Res., B*, 1940, **18**, 405)

followed by the Emmerie and Engel reaction with iron dipyridyl (*cf. Rec. Trav. Chim. Pays-Bas*, **1938**, **57**, 1351) gives low and erratic results for total tocopherols. An investigation has shown that the interfering substance responsible for such results can be extracted with 5% KOH soln. from a hexane soln. of the oil; subsequent treatment of a benzene soln. of the oil with diatomaceous earth reduces impurities still further. Aliquots of the purified oil are then evaporated under nitrogen, the residue is dissolved in 1 ml of hexane, and 1 ml of $FeCl_3$ soln. (0·1% ethanolic) is added, followed within 30 sec. by 1 ml of 2:2′-dipyridyl soln. (0·25% ethanolic). After 30 sec., 7 ml of ethanol is added, the mixture is set aside for 2 min., then transferred to a cuvette and the extinction at 520 mμ is measured after a further 2·5 min. Results are referred to a calibration curve. G. HARDING

See also Abstracts—**1907**, Determination of vanillin. **1980**, Determination of 7-oxocholesterol in wool wax. **2026**, Determination of dimefox residues in hops.

Sanitation

2001. **Rapid method of determining nickel carbonyl in air.** V. S. Fikhtengol'ts and N. P. Kozlova (Leningrad Inst. of Work Hyg. and Occupational Diseases). *Zavod. Lab.*, 1957, **23** (8), 917.—To determine $<0{\cdot}002$ mg of $Ni(CO)_4$ per litre, 1 litre of air is passed for 1 min. through 2 ml of a 1·5% soln. of iodine in CCl_4. After treatment with Na_2SO_3 the soln. is analysed colorimetrically for Ni. With lower contents of $Ni(CO)_4$, a larger vol. of air and another absorption vessel are used. G. S. SMITH

2002. **The use of ion exchangers in analysis of water.** M. Pavlík and M. Mach (Inst. Technol. of Water, High-School Chem. Technol., Prague). *Voda*, 1957, **36** (4), 105–107.—A survey is presented, with a description of the determination of mineral acids in water. J. ZÝKA

2003. **The influence of iron on the determination of oxygen in water by Winkler's method.** K. Buriánek (ČKD-Dukla, Prague). *Voda*, 1957, **36** (8), 211–212.—Errors due to Fe in Winkler's iodimetric method can be avoided by maintaining accurately the interval between acidifying the sample, the final titration and the blank. J. ZÝKA

2004. **Influence of chloride in the dichromate-value test.** W. M. Cameron and T. B. Moore (Middx. County Council, Main Drainage Dept., Mogden Works, Isleworth, England). *Analyst*, 1957, **82**, 677–682.—The method of Moore *et al.* (*Brit. Abstr. C*, 1949, 415; 1952, 68) for the determination of oxygen absorbed by polluting matter in water by heating the sample under reflux with acid $K_2Cr_2O_7$, with Ag_2SO_4 as catalyst, was examined and was found to give results with pure soln. much nearer to the theoretical value than those of the conventional $KMnO_4$ method, but the presence of Cl^- causes some difficulties. If Ag_2SO_4 is added before the Cl^- are oxidised, the pptd. AgCl is difficult to oxidise, Cl^- present are not completely oxidised and the correction for unoxidised Cl^- is not precise. Correction is more difficult when Cl^- and N occur together because the nitrogenous matter is oxidised by the free Cl liberated. It is recommended that sufficient $N/8$ $K_2Cr_2O_7$ soln. should be added to oxidise the Cl^- in addition to the normal 25 ml required for the sample, and that the mixture should be heated under reflux for a time before addition of the Ag_2SO_4. Approx. corrections for use in the presence of Cl^- and of Cl^- and N together are developed. A. O. JONES

2005. **Quantitative determination of traces of carbon dioxide in water.** P. P. Jennings and E. M. Osborn (Centr. Electr. Authority, Skelton Grange Power Sta., Leeds, England). *Analyst*, 1957, **82**, 671–676.—By means of the apparatus described and illustrated, the CO_2 in the acidified sample is aspirated for 1 hr. with CO_2-free air into 0·1 *N* NaOH soln., which is then titrated with 0·01 *N* CO_2-free H_2SO_4. The titre between pH 8·3 and 5·0 measured with a glass electrode system indicates the amount of CO_2 in the sample. Results show that the method is capable of determining CO_2 in boiler water with an accuracy of $\pm 0{\cdot}003$ mg per litre in a 10-litre sample provided that the sample contains >1 mg of CO_2. A. O. JONES

2006. **Photometric determination of aluminium in water with aluminon.** V. Zdeněk (ČKD-Dukla, Prague, Czechoslovakia). *Voda*, 1957, **36** (2), 35–36.—The influence of Fe in this determination was studied and it was found that thioglycollic acid was a suitable masking agent. *Procedure*—To the sample (100 ml) add thioglycollic acid (1% soln.) (0·2 ml), mix, and add 2 ml of the reagent containing aluminon (0·05 g), acacia gum (1 g), ammonium acetate (26·7 g), conc. HCl (25 ml) and H_2O (170 ml). Adjust the mixture to pH 4 with acetate buffer soln., heat on a water bath (4 min. exactly), then set aside for 1·5 hr. and measure the colour at 546 mμ and 20°. J. ZÝKA

2007. **Accurate determination of sulphates in waters by direct titration.** M. Le Peintre and J. Richard. *Chim. Anal.*, 1957, **39** (9), 331–333.—The method of Fritz and Freeland (*cf. Anal. Abstr.*, 1955, **2**, 609) is improved by the addition of an aq. suspension of $BaSO_4$ to initiate and accelerate the pptn. of SO_4^{2-} during titration. *Procedure*—To the sample ($\simeq 50$ ml) add 96% ethanol (40 ml), acetate buffer (1 ml), aq. $BaSO_4$ suspension (2 ml), and 0·2% (w/v) aq. alizarin red S soln. (2 to 3 drops). Titrate the mixture with 0·005 *M* $BaCl_2$ or $Ba(ClO_4)_2$ at pH 3·2, adding the titrant in small amounts and shaking vigorously. The end-point is sharp. Interfering ions can be removed by passing the sample through a cation-exchange column, but PO_4^{3-} should be removed by pptn. with $MgCO_3$. The error ranges from $\pm 10\%$ to $\pm 1\%$ for concn. of SO_4^{2-} increasing from 5 μg to 50 μg per ml. W. J. BAKER

2008. **Volumetric determination of sulphates in natural waters.** G. N. Nechiporenko. *Gidrokhim. Materialy*, 1957, **26**, 207–217; *Ref. Zhur., Khim.*, 1957, Abstr. No. 57,824.—In determining sulphates in natural waters, the use of direct titration with a soln. of $Pb(NO_3)_2$ in an aqueous ethanolic medium in the presence of diphenylcarbazone as indicator is recommended. The optimum pH value is 4·0 to 6·6. C. D. KOPKIN

2009. **Catex method for the determination of sulphates in water and the comparison with commonly used methods.** M. Pavlík and M. Mach (Inst. Technol. of Water, High-School Chem. Technol., Prague). *Voda*, 1957, **36** (5), 123–126.—By analysing numerous samples of water it was found that, when determining sulphates, the commonly

used titrimetric chromate method yields inaccurate results and the complexometric method is suitable only when the approximate content of sulphates is known. The new method proposed, in which a column with Catex (cation-exchange resin) is used, is suitable for all samples containing >10 mg of SO_4^{2-} per litre. J. ZÝKA

2010. **Field determination of uranium in natural waters.** F. N. Ward and A. P. Marranzino. *U.S. Geol. Survey Bull.* No. 1036–J, 1957, 181–192.—The sample is acidified with HNO_3, Na_2HPO_4 is added to give a pH of 5·9 to 6·1, and the uranium phosphate complex is collected on paper pulp. The complex is broken up with $Al(NO_3)_3$, separated by paper chromatography, and treated with $K_4Fe(CN)_6$. Uranium is determined by comparing the resulting brown band with standard bands. The method is reasonably precise for U in concn. of 2 to 200 p.p.m. CHEM. ABSTR.

2011. **Detecting the presence of halogens in water.** Central Electricity Authority and E. C. Potter. Brit. Pat. 790,419; date appl. 15.9.54.—Two electrodes connected together outside the water, through a resistor of high ohmic value, are immersed in the water. The electrodes are of two different metals, *e.g.*, platinum and tungsten, and cause an increased current to flow through the resistor when a halogen is present in the water as compared with the current when no halogen is present. The current through the resistor is determined by measuring the voltage drop across it. J. M. JACOBS

2012. **Chromatographic identification and determination of organic acids in water.** H. F. Mueller, T. E. Larson and W. J. Lennarz (Illinois State Water Survey, Urbana, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 41–44.—The organic acids in aq. soln. are adsorbed on silicic acid and are subsequently eluted from the column by various concn. of butanol in $CHCl_3$. The acidity of the fractions is determined titrimetrically with NaOH; each acid has a characteristic peak eluate volume. Recoveries of the 11 acids tested were $>\simeq 90\%$. The technique also provided a new method for the separation of anionic synthetic detergents from analytical interferences. The method was applied to the analysis of river water, sewage, and anaerobic digestion liquors, and preliminary data for various river waters showed a somewhat uniform correlation in the types of acid appearing. G. P. COOK

2013. **Determination of long-lived fall-out in rain-water.** R. G. Osmond, A. G. Pratchett and J. B. Warricker (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2165, 1957, 21 pp.—Methods are given for the determination of ^{89}Sr, ^{90}Sr, ^{137}Cs and ^{144}Ce. The sample (1 to 30 litres) is collected in a polyethylene vessel containing HNO_3 and carriers for Sr, Cs and Ce. After evaporation to $\simeq 20$ ml, the soln. is filtered, and standard radiochemical separations of the three elements are performed; special steps are necessary to ensure separation of the Sr from any Ca and sulphate in the rain-water. The residue from the filtering is analysed only if a gross β-count shows that it could contain a significant fraction of the activities. β-Counting equipment with a low background (1 to 1·2 counts per min.) is used in the assay of the less active samples. G. J. HUNTER

2014. **Recommended methods for the analysis of trade effluents. Determination of oxygen demand.** The Joint A.B.C.M. - S.A.C. Committee on Methods for the Analysis of Trade Effluents. *Analyst*, 1957, **82**, 683–708.—The use of $K_2Cr_2O_7$ in boiling acid soln. for determining the C.O.D. of effluents (the dichromate value) has considerable advantages over the $KMnO_4$ method, being more reproducible and applicable to a wider variety of samples. Detailed descriptions are given of the determination of oxygen demand by the $K_2Cr_2O_7$ method and by the $KMnO_4$ method, the determination of dissolved oxygen by the Winkler method and its modifications, *viz*, the Alsterberg (or sodium azide) and the Rideal - Stewart (or permanganate) modifications, and the determination of B.O.D. by the dilution method and the manometric method. The effects of some inhibitory substances on the determination of B.O.D. are tabulated. Special procedures are suggested for samples containing polythionate, thiosulphate, sulphite, sludges and activated sludge. (62 references.) A. O. JONES

2015. **Recommended methods for the analysis of trade effluents. Methods for the determination of antimony, barium soluble in dilute hydrochloric acid and cadmium.** The Joint A.B.C.M. - S.A.C. Committee on Methods for the Analysis of Trade Effluents. *Analyst*, 1957, **82**, 764–767.—After destruction of organic matter, the residue is treated with a HBr - Br mixture and dil. HCl, Br is removed by boiling, the filtered soln. is evaporated to a specified vol. and As is removed by pptn. with NaH_2PO_2. From the filtrate, Sb_2S_3 is pptd. by H_2S, collected and dissolved in a hot mixture of HCl, H_2O_2 and bromine water. After removal of Br by boiling, H_2SO_3 is added and, after 1 hr., the SO_2 is removed by boiling. The hot soln. is then titrated with standard $KBrO_3$ soln. Barium sol. in dil. HCl is pptd. under controlled conditions from the neutralised sample with Na_2SO_4 soln. in the presence of EDTA (to prevent interference by Pb and Sr) and glycerol. The turbidity is measured absorptiometrically against water and referred to a calibration graph prepared with standard $BaCl_2$ soln. Cadmium is measured as its red complex with dithizone in CCl_4, interference from Cu and Ni being prevented by complexing with KCN. The extinction is measured spectrophotometrically at 525 mμ or absorptiometrically with a green filter and referred to a calibration graph. For contents of Cd $\gg 25$ μg, the colour may be estimated visually against standards. A. O. JONES

2016. **Simple arrangement for the polarographic determination of dissolved oxygen in industrial wastes.** D. Klíma (Res. Inst. for Meat and Fish, Brno, Czechoslovakia). *Voda*, 1957, **36** (6), 148–151.—The equipment comprised a sensitive galvanometer, a Kohlrausch drum and a dropping mercury electrode. In contrast with other commonly used methods, nitrites do not interfere in this determination. The max. of the polarographic wave should be measured at $-1{\cdot}6$ V. J. ZÝKA

2017. **Determination of oil in refinery effluent waters. Report of the subcommittee on refinery effluent water analytical methods, Committee on Analytical Research, Div. of Refining, A.P.I.** American Petroleum Institute. *Anal. Chem.*, 1958, **30** (1), 36–40.—A co-operative study of three procedures for the determination of total oily matter in refinery effluent water was undertaken. An i.r. technique and a direct extraction - pycnometer

procedure were compared with one of two approved A.P.I. methods. The results showed that the i.r. procedure was generally the best, recoveries being $> \simeq 90\%$. The direct extraction - pycnometer procedure had certain advantages, particularly when volatile materials were present and the oil concn. was low. Both procedures were superior to the A.P.I. approved method. Comprehensive data for all methods are tabulated. G. P. Cook

See also Abstracts—**1793**, Determination of boron in water. **1829**, Determination of sulphate in rainwater.

Agriculture and Plant Biochemistry

2018. **Quantitative micro-analysis of leaf tissue for certain organic constituents with special reference to sampling error.** K. P. Lamb (Plant Diseases Div., D.S.I.R., Auckland). *N.Z. J. Sci. Tech., B*, 1957, **38** (9), 1009–1017.—Procedures are given for the quant. determination of total N, amino-N fractions and sol. carbohydrates in microgram amounts in discs of leaf tissue weighing approx. 5 mg (fresh) or 1 mg (dry). Statistical aspects of sampling and analytical error are considered. I. Jones

2019. **Estimation of vanadium in plant materials.** G. B. Jones (Div. of Biochem. and Gen. Nutr., C.S.I.R.O., Univ. Adelaide, Australia). *Anal. Chim. Acta*, 1957, **17** (2), 254–258 (in English).—Microgram or less amounts of V in the presence of Mo and W are determined polarographically, between $-0{\cdot}6$ and $-1{\cdot}3$ V *vs.* the anode pool, in an electrolyte that is 6·5 *M* to aq. NH_3 and 3 *M* to NH_4Cl. Iron, Cu, Cr and Zn are removed electrolytically at a mercury cathode, whilst V, Ti, Mo and W are separated from Mn by extraction with cupferron in $CHCl_3$. Separation from Ti is effected by extraction of V, Mo and W with Na diethyldithiocarbamate in $CHCl_3$ from a soln. buffered at pH 4·5 to 5·0. Although there is a long chemical separation, the recovery of V is 90 to 95%. J. H. Waton

2020. **Carrier precipitation of trace elements. Radio-isotope evaluation of efficiency.** E. E. Pickett and B. E. Hankins (Univ. of Missouri, Columbia, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 47–50.—The efficiency of separation of the trace elements, Cu, Co and Mo, from the major constituents of agricultural samples was determined by pptn. with aluminium oxinate or indium oxinate carrier, with or without the addition of thionalide or tannic acid. All three elements can be pptd. with the three reagents together. With aluminium oxinate alone, Cu is incompletely recovered, with indium oxinate alone no element is completely recovered. Experiments giving information on the mechanism of the co-pptn. are described. E. G. Cummins

2021. **The use of paper chromatography in the separation of the valency states of biologically important metals [in the metabolism of micro-organisms].** H. M. Stevens (Long Ashton Res. Sta., Bristol, England). *J. Sci. Food Agric.*, 1957, **8** (Suppl. issue), s38–s43.—Separations of the valency states of Mo, V, Co, Fe, Mn and Cu were obtained by downward chromatography on acid-washed Whatman No. 1 filter-paper strips. Eluents giving optimum resolution for a system were obtained by mixing a "lipophilic" liquid (*e.g.*, an alcohol or ketone) with a "hydrophilic" one (*e.g.*, water). Enhancement of the former property was obtained by replacement of the alcohol or ketone by a higher homologue or (to a greater degree) by an ether. The addition of acids or of salts in various concn. similarly increased the "hydrophilic" properties. The separations of Mo^{VI} and Mo^{V} as thiocyanates and Fe^{III} and Fe^{II} as acetates appeared to be quantitative. D. F. Phillips

2022. **Determination of aluminium in soil extracts by means of its depressing action on the flame emission of calcium.** M. Pinta and H. Aubert. *Compt. Rend.*, 1957, **244** (7), 873–876.—An alkaline extract of the soil is examined quantitatively for Fe and Ti, and an aliquot of the extract is diluted with a known vol. of standard Ca^{2+} soln., leaving the final concn. 200 and 1000 μg of Ca^{2+} and Fe^{3+} per ml, respectively. The soln. is subjected to flame photometry and the depression of the Ca 622-mμ emission is measured: the concn. of Al^{3+} is derived from calibration curves, which are not linear, allowance being made for the relatively small effect of Ti^{4+}. A precision of $\pm 5\%$ over the concn. range 25 to 250 μg per ml is quoted. D. A. Pantony

2023. **The effect of colloidal organic matter on the precipitation of barium sulphate and a modified method for determining soluble sulphate in soils.** P. R. Hesse (E. African Agric. and Forestry Res. Organisation, Kikuyu, Kenya). *Analyst*, 1957, **82**, 710–712.—For the routine determination of SO_4^{2-} in soils the method of Chesnin and Yien (*Brit. Abstr. C*, 1952, 223) is probably the most convenient and gives the most reproducible results, but the acetic acid - Na acetate extracting buffer soln. also extracts organic matter. This acts as a protective colloid with low concn. of SO_4^{2-}, thereby causing low results, and is co-pptd. with higher concn., thereby causing high results. In the modification now described, $FeCl_3$ soln. is added to the sample extract and the colloidal organic matter is co-pptd. with $Fe(OH)_3$ by addition of NaOH soln. and removed by filtration before addition of $BaCl_2$ soln. Glacial acetic acid is added to the filtrate and the SO_4^{2-} are then pptd. as $BaSO_4$. The determination is completed as in the method of Chesnin and Yien (*loc. cit.*). Results indicate that the errors due to colloidal protection and co-pptn. of organic matter are eliminated. A. O. Jones

2024. **Spectroscopic analysis of chelate solutions; application to iron chelates in soils and plants.** D. G. Hill-Cottingham (Long Ashton Res. Sta., Bristol, England). *Soil Sci.*, 1957, **84**, 43–49.—Iron chelate soln. show max. absorption at wavelengths at which neither the chelating agent nor the calcium chelate has any influence. The extinction of such soln. is a measure of the iron chelate present provided that the pH of the soln. lies within specified limits. Optimal wavelengths (mμ) and pH ranges determined were—EDTA, 258, 2·0 to 6·0; N-hydroxyethylethylenediaminetriacetic acid, 262, 2·0 to 3·0; NN′-dihydroxyethylethylenediaminediacetic acid, 250, 2·7 to 2·9; diethylenetriaminepenta-acetic acid, 260, 3·0 to 7·0; *cyclo*hexane-1:2-diaminetetra-acetic acid, 258, 2·0 to 9·0; and ethylenediaminedi-(*o*-hydroxyphenylacetic acid), 485, 4·0 to 10·0. Chelates other than that of Fe may be determined by measuring the increase in extinction on addition of iron salts in acid soln. (pH about 3·0). Under these conditions other metal chelates are converted into iron chelates. A. G. Pollard

2025. **Determination of small quantities of nitrite in fish meal.** R. Seibold (Württ. Landesversuchanst. Landw. Chem., Stuttgart-Hohenheim, Germany). *Landw. Forsch.*, 1957, **10**, 50–55.—Weigh a 10-g sample into a 250-ml Stohmann flask. Add about 100 ml of H_2O and 1 ml of 2 *N* NaOH, shake for 30 min., dilute with water to the mark, and filter. Transfer 50 ml of the filtrate to a 100-ml flask, add 10 ml of 2 *N* NaOH, and slowly add an equiv. vol. of 20% $AlCl_3$ soln. Shake, filter, transfer an aliquot of the colourless filtrate to a 50-ml flask, add ≃20 ml of water followed by 25 ml of test soln., and dilute immediately with water to the mark. After 30 min. measure the extinction at 525 mμ. The test soln. contains 0·4 g of *p*-nitroaniline and 0·7 g of 1-naphthylamine in 100 ml of methanol, and must be stored in the dark. Mix 8 ml of this soln. with 50 ml of 10 *N* HCl and dilute to 1 litre.

CHEM. ABSTR.

2026. **Determination of dimefox residues in hops.** K. Field and E. Q. Laws (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1957, **82**, 667–671.—The dimefox (NNN′N′-tetramethylphosphorodiamidic fluoride) is extracted from dry hops with methanol according to a specified procedure and, after addition of petroleum spirit and ice-cold water, the aq. layer is separated and extracted with chloroform. The chloroform extract is evaporated and the residue is dissolved in ether. The ethereal soln. is applied to a chromatographic column of MgO and the eluate is collected, elution being continued with ether. After removal of the ether the residue is dissolved in water, treated with *N* aq. $HClO_4$, maintained at 25° ± 2° for 20 min. and then extracted with chloroform, the extract being discarded. The aq. layer is evaporated to fuming point with HCl and HNO_3, then boiled with aq. NH_3. The soln. is treated with an ammonium molybdate reagent and extracted with an *iso*butyl alcohol - benzene mixture, and the separated organic layer is washed with ethanolic H_2SO_4 and then with a dil. $SnCl_2$ soln. The extinction of the blue organic layer is measured at 730 mμ against a solvent blank (*iso*butyl alcohol - benzene - ethanolic H_2SO_4) and referred to a calibration graph. Recovery is 85 to 95% for amounts of ≃0·5 μg in 10 g of hops. A. O. JONES

2027. **Colour reaction of 2:6-dibromo-*p*-benzoquinone-4-chlorimine with thiophosphate insecticides on paper chromatograms.** J. J. Menn, W. R. Erwin and H. T. Gordon (Dept. of Ent. and Parasit., Univ. of Calif., Berkeley, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (8), 601–602.—A simple chromatographic method for determining phosphate ester insecticides is described. The developed chromatogram is sprayed with a soln. of the reagent (0·5% in *cyclo*hexane) and heated in an oven at 110° for 7 min. Spot colours range from yellow to brownish red; as little as 1 μg of some insecticides can be detected. S.C.I. ABSTR.

2028. **Field persistence of the acaricide 4:4′-dichloro-α-(trichloromethyl)benzhydrol [2:2:2-trichloro-1:1-di-(*p*-chlorophenyl)ethanol] (FW-293) on and in mature lemons and oranges.** F. A. Gunther, R. C. Blinn, L. R. Jeppson, J. H. Barkley, G. J. Frisone and R. D. Garmus (Univ. of Calif. Citrus Expt. Sta., Riverside, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (8), 595–598.—Residues of this acaricide persisting on and in lemons and oranges and in dried citrus cattle feed were determined; the half-life values were 170 to 350 days for Valencia oranges and 120 to 150 days for lemons. There was little metabolic or other degradation of FW-293 as indicated by results of three methods of determining amounts of residue, *viz*, total chloride, total ketone and colorimetric method. Negligible amounts were found in the edible portion. The peel retained ≃30% of FW-293 residues after being processed into dried cattle feed. S.C.I. ABSTR.

2029. **Estimation of parathion residues in the presence of azobenzene.** W. H. Read, J. T. Hughes and R. J. Smith. *Annu. Rep. Glasshouse Crops Res. Inst.*, 1954–55, [1957], 52–54.—The method is based on benzene extraction and aq. alkali hydrolysis, followed by reduction of the *p*-nitrophenol by zinc dust and HCl, and conversion into a blue indophenol compound with *o*-cresol and ammonia. This reaction appears to be specific for *p*-nitrophenol in the presence of azobenzene. E. G. BRICKELL

2030. **Determination of ethylene oxide in fumigated copra products.** J. H. Benedict (Procter and Gamble Co., Miami Valley Lab., Cincinnati, Ohio). *J. Amer. Oil Chem. Soc.*, 1957, **34** (9), 450–451.—Ethylene oxide (**I**) reacts with the HBr in an acidified soln. of $MgBr_2$ to form 2-bromoethanol. The decrease in acidity of the $MgBr_2$ is proportional to the **I** absorbed. Meter 1·5 litres of air successively through a wash-bottle containing 40% aq. NaOH soln., a trap wash-bottle, a 3-necked 3-litre flask containing 400 g of copra, a vertical condenser, a safety tube, the flowmeter, and 2 absorption tubes each containing 20 ml of $MgBr_2$ soln. (500 g in 1 litre of 0·02 *N* H_2SO_4). After 1 hr., stop the air flow, add 2 litres of freshly boiled hot distilled water to the copra, mix thoroughly, keep the flask hot, and resume the air passage for 1 hr. Titrate the contents of the absorption tubes with 0·02 *N* NaOH to bromocresol green indicator, while stirring with a magnetic stirrer. Determine the blank. The recovery of known additions of **I** was > 90%. The lower limit of detection is 0·3 p.p.m.

G. HELMS

See also Abstracts—**1815**, Determination of arsenate. **1985**, Determination of DDT in cereals. **1996**, Determination of vitamin A in poultry mashes.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

2031. **Improvements in or relating to sonic gas analysers.** C. A. Parsons & Co., Ltd. [Inventors: A. E. Martin, D. Mounfield and J. Smart]. Brit. Pat. 788,801; date appl. 11.3.55.—The analyser comprises one or more containers packed with a material (grains of polyethylene, metal granules, glass beads) having interstices which provide continuous paths for the gas, means for transmitting sonic or ultrasonic vibrations through the gas in the interstices and for receiving them after transmission through the gas, and means, responsive to changes in the velocity of the vibrations, for detecting changes in the composition of the gas in the container. J. M. JACOBS

2032. **Vacuum drying ovens for microchemical purposes. Microchemical apparatus. Group G: Heating, cooling and drying accessories.** British Standards Institution (2 Park St., London). B.S.

1428:Part G2:1957, 11 pp.—Requirements are specified for vacuum ovens suitable for drying substances in microchemical vessels.
O. M. WHITTON

2033. **Improvements in and relating to instruments for indicating or measuring moisture content of materials.** J. Lyons & Co., Ltd. [Inventor: L. G. Toplis]. Brit. Pat. 789,123; date appl. 1.11.54 (addn. to Brit. Pat. 687,472, dated 9.9.48).—In the instrument described in B.P. 687,472 the material under test is inserted in a cell having electrodes forming a condenser. The moisture content of the sample is ascertained from the change in capacity caused by the insertion of the material between the electrodes. For determining the moisture content of bulk materials, *e.g.*, bales of jute, without unpacking them, the electrodes comprise a pair of vertical spaced plane plates, each mounted above and spaced from an insulating floor by a frame of insulating material. A truck is provided for supporting the bale and moving it progressively between the plates. J. M. JACOBS

2034. **Modifications of Beckman Aquameter to improve precision and flexibility of operation.** E. H. Unger and A. G. Herzog (Socony Mobil Oil Co., Inc., Paulsboro, N.J., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 157–158.—The versatility, rapidity and accuracy with which the water content of a wide variety of samples can be determined with the Beckman Model KF-3 Aquameter have been increased by making easy and inexpensive changes in the reagent-delivery system and titration vessel. No alterations have been made in the end-point detection and indication system. K. A. PROCTOR

2035. **Density - composition tables for aqueous solutions of nitric acid.** British Standards Institution (2 Park St., London). B.S. 975:1957, 56 pp.—In this revised Standard the text has been amended to accord with the new B.S. on hydrometers. By the use of the tables with the appropriate hydrometers, the concn. of any given aq. soln. of HNO_3 can be determined, or a soln. of known concn. can be made up. The temp. range 10° to 40° is covered.
O. M. WHITTON

2036. **Ultra-micro method for molecular-weight determination.** G. O. Guerrant (Shell Development Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 143–149.—Molecular weights can be determined with an accuracy of 3%, with less than 1 mg of sample, by means of a modified isothermal distillation method in which the sample is suspended from a calibrated helical tungsten spring enclosed within a vessel containing a standard reference soln. The temp. of the equilibrium system must be controlled for long periods to within ±0·005°. Three days are required for a determination, but the operator time per single determination is 2 hr. The method is not applicable to materials boiling below 275°.
K. A. PROCTOR

2037. **Filtration apparatus for microchemical analysis. Microchemical apparatus. Group F: Filtration accessories.** British Standards Institution (2 Park St., London). B.S. 1428:Part F1: 1957, 15 pp.—Requirements are specified for filter-crucibles, filter-sticks and filter-tubes of materials and sizes suitable for general use in microchemical analysis. Ancillary equipment suitable for use with B.S. filter-sticks is described. Tests for constancy of wt. of platinum, silica, porcelain and glass apparatus, respectively, are given.
O. M. WHITTON

2038. **Device for measuring R_F values.** R. L. Clements (Dept. of Agric. Biochem., Ohio State Univ., Columbus, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 160–161.—The device is easily constructed from aluminium or any such rigid material, and a high degree of precision can be attained without special machining or engraving. K. A. PROCTOR

2039. **The filling of adsorption columns.** N. Békésy (Heilpflanzen-Forsch. Inst., Budapest, Hungary). *Z. anal. Chem.*, 1957, **157** (4), 272–274.—A method for filling chromatography columns and removing the separate sections is described.
E. G. CUMMINS

2040. **Balance-type fraction collector.** A. Kočent, Z. Brada and B. Keil (Chem. Inst., Acad. Sci., Prague). *Chem. Listy*, 1957, **51** (8), 1575–1576.—A new variation of the balance-type fraction collector avoids the necessity of using test-tubes of exactly the same weight when separating chromatographically on columns. J. ZYKA

2041. **Water-curtain enclosure for spraying paper chromatograms.** E. C. Fiebig and H. Siegel (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 161.—The portable enclosure described is particularly useful when reagents that leave noxious or dangerous residues are used. The reagents are continuously removed by dilution by a flowing film of water on all surfaces of the enclosure exposed to the spray. The enclosure has proved effective for a variety of reactive and intensely coloured reagents, both aqueous and non-aqueous. Water and drainage are the only services required for operation. K. A. PROCTOR

2042. **A chromatographic chamber for one-dimensional ascending and descending paper chromatography.** C. Kowala (C.S.I.R.O., Div. of Ind. Chem., Melbourne, Australia). *Chem. & Ind.*, 1957, (37), 1234–1235.—The apparatus and procedure are described. G. S. ROBERTS

2043. **Process and apparatus for paper chromatography and paper electrophoresis.** Schering A.-G. Brit. Pat. 791,570; date appl. 25.5.56. Germany, date appl. 26.5.55.—The normal processes of chromatography or electrophoresis are carried out with rolled, instead of extended, paper strips, the individual layers of the roll being prevented from coming into contact with one another by means of an interleaving foil of synthetic resin of low wettability by the solvent used, *e.g.*, a F-containing polymer. J. M. JACOBS

2044. **Paper chromatography of unstable substances.** K. Schwarz and A. A. Bitancourt (Plant Cancer Res. Centre, Inst. Biol., São Paulo, Brazil). *Science*, 1957, **126**, 607–608.—The procedure recommended is termed "double chromatography," and comprises two-dimensional chromatography in which the same solvent is used in both directions. The method is illustrated by chromatograms of indolylpyruvic acid. H. F. W. KIRKPATRICK

2045. **New aid to paper chromatography.** P. H. List (Inst. für Pharm. und Lebensmittelchemie der Univ. Würzburg, Germany). *Naturwissenschaften*, 1957, **44** (9), 280–281.—The apparatus assists in obtaining small circumscribed spots of the test substance on the paper and also serves as an extraction apparatus. A small funnel is fused into the floor of a small beaker (4·5 × 4 cm) in such a way that the rim of it projects a short distance above the floor level. The mouth of the beaker carries a standard 29-joint and connects with a short tube into which a cooling finger has been fused. The exit

of the funnel is drawn into a fine capillary tube into which a small tuft of cotton wool is inserted. Solvent (water, organic solvent) is poured into the floor of the beaker, is evaporated by a current of warm air playing on the beaker, condenses on the cold finger and drips into the funnel where it dissolves or extracts the material to be chromatographed. The tuft of cotton wool makes contact with the sheet of chromatography paper to which the material is thus transferred. The current of warm air is so directed that it dries the spot on the paper as well as warming the beaker. Even with water the spots on the paper do not exceed 1 cm in diam.

E. KAWERAU

2046. **Process for the continuous chromatographic separation of mixtures.** J. J. Solms. British Patent 786,896; date appl. 3.2.55. Switzerland, date appl. 3.2.54.—The mixture is applied to a separating medium (hollow cylinder of filter-paper or annular bed of ion-exchange resin) so that a uniform relative movement occurs between the point of application and the separating medium, *e.g.*, by rotating the cylinder. An elution agent is passed continuously, under gravity, through the medium, and the individual separated components are continuously collected. J. M. JACOBS

2047. **Two-stage gas - liquid chromatography.** M. C. Simmons and L. R. Snyder (Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 32–35.—A two-stage gas - liquid chromatographic apparatus is described in which a non-polar column is connected to a polar column in such a manner that preliminary cuts prepared by the first column can be either rejected or charged directly to the second column. Interfering low- and high-boiling components can therefore be vented from the first column and only the cuts in the desired narrow boiling-range are admitted to the second column for analysis. Application to the analysis of Platformer stream samples for the key components involved in C_6 conversion, namely methyl*cyclo*pentane, *cyclo*hexane and benzene, gave results in close agreement with those obtained by a mass-spectrometric procedure. Data for 30 C_5 to C_8 compounds are listed. G. P. COOK

2048. **Improvements in or relating to gas chromatography.** Sir H. Grubb Parsons & Co., Ltd. [Inventor: A. E. Martin]. Brit. Pat. 790,217; date appl., 18.10.54.—The components of a gas mixture which have been separated from one another by gas chromatography are detected and quant. analysed by conveying the component gases through an oxidising region (*e.g.*, passing org. gases over heated CuO so that the carbon is converted into CO_2) and thence to an infra-red analyser equipped with a detecting condenser sensitive to the oxidised gas. J. M. JACOBS

2049. **Fraction cutter for gas chromatography.** A. Weinstein (Radiol. Warfare Div., U.S. Army, Dugway Proving Ground, Utah, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1899–1900.—The cutter described is compact and simple to operate. It will separate a normal sized sample of two components in a state of purity such that contamination of one fraction by the other cannot be detected by gas chromatography or mass spectrometry.

K. A. PROCTOR

2050. **A semi-micro respirometer.** J. W. Ridge (Inst. of Ophthalmol., Judd St., London). *Biochem. J.*, 1957, **67** (2), 177–179.—A micro-Barcroft apparatus is described in which the reaction and compensation vessels are of different capacities and in which the pressures on either side of the manometer are equilibrated by altering the vol. on the compensating side by means of a micrometer syringe. In this way pressure changes in the reaction vessel are magnified approx. in the ratio of the capacities of the two chambers and can be estimated with a smaller error. J. N. ASHLEY

2051. **Automatic direct-reading apparatus for determining the surface area of powders.** F. J. Spillane (Lafarge Aluminous Cement Co. Ltd., West Thurrock, Essex, England). *Analyst*, 1957, **82**, 712–715.—The apparatus described and illustrated measures, in effect, the permeability to air of a powder bed of fixed depth. The air is introduced into a reservoir by means of a blow bulb until its pressure depresses the mercury in a U-tube gauge to a point below an electrical contact sealed into one limb, a similar contact at a higher point in the other limb being then immersed in the mercury. As the air is allowed to permeate through a bed of the powder the mercury rises to the lower contact, thereby starting a clock motor. After an interval T (sec.) the mercury sinking in the other limb breaks contact and thus stops the clock. Conditions can be arranged so that T is the only variable, and the specific surface will then be proportional to $\sqrt{T}$. Calibration of the apparatus to provide a direct-reading scale is described. A. O. JONES

2052. **Variable-temperature Dewar-type tubular desiccator.** G. F. Smith (Univ. Illinois, Urbana, U.S.A.). *Anal. Chim. Acta*, 1957, **17** (2), 192–194 (in English).—A substitute for the drying pistol is described, which is more versatile and more efficient. The apparatus comprises a jacketed horizontal tube, fitted at each end with stopcocks. Heating to a selected temp. is achieved by boiling suitable liquids and allowing the vapours to circulate through the jacket. J. H. WATON

See also Abstracts—**1763**, Apparatus for gradient elution chromatography. **1913**, Liquid substrates in gas chromatography.

Optical

2053. **A simple fan-scale.** E. R. Stuart (Univ. Chem. Lab., Trinity Coll., Dublin). *Chem. & Ind.*, 1957, (37), 1235.—The intervals on the linear wave-number scale of a Hilger H.800 i.r. spectrophotometer are variable between wide limits. The paper describes the construction of a fan-shaped scale, by a simple photographic procedure, for dividing these scale intervals into 50 equally spaced units.

G. S. ROBERTS

2054. **Glass pens for Beckman Model DK-2 spectrophotometer.** A. C. Arcus (Med. Sch., Dunedin, N. Zealand). *Anal. Chem.*, 1958, **30** (1), 159–160.—The use of readily interchangeable glass pens enables spectra to be recorded in different colours. The position of the pen point on the chart is accurately reproduced when pens are changed so that there is $<0{\cdot}1\%$ variation on the transmittance scale. K. A. PROCTOR

2055. **Improvements in and relating to spectrometers.** C. A. Parsons & Co. Ltd. [Inventor: A. E. Martin]. Brit. Pat. 790,928; date appl. 21.6.55.—The plane-ruled diffraction grating of the spectrometer is rotated by means of a screw so that an exact linear relationship is obtained between the turns of the screw and the reciprocal of the wavelength or wave-number. Motion is imparted to the

grating by a lever arm actuated by a member which is constrained to move by the action of the screw. This member has that part of its periphery which is in contact with the lever arm of circular contour with a radius of curvature equal to $50\,h/d\,\cos\,\delta/2$, where d is the spacing of the grating in microns, δ is the difference between the angle of diffraction and the angle of deflection, and h is the displacement of the end of the screw corresponding to 100 wave numbers in the first order on the wave-number scale. J. M. JACOBS

2056. **High-temperature cell for infra-red spectroscopy.** A. L. Olsen (U.S. Ordnance Test Sta., China Lake, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 158.—The cell is designed for use in the Perkin-Elmer Model 21 spectrophotometer, and can be used up to 300°. K. A. PROCTOR

2057. **Fluorescence spectrum attachment for Beckman DU spectrophotometer.** J. A. McCarter (Dalhousie Univ., Halifax, Canada). *Anal. Chem.*, 1958, **30** (1), 158–159.—In this attachment a cuvette containing the fluorescent sample is mounted in the position normally occupied by the hydrogen lamp. The sample is excited by illumination with filtered u.v. radiation through a hole in the lamp backplate. The fluorescent sample thus becomes the light source of the spectrophotometer which, when fitted with a photomultiplier detector, enables the fluorescent spectra of very dilute samples to be measured. A soln. of quinine sulphate in 0·1 N H_2SO_4 containing 0·3 μg per ml gave a reading of 52·9 $\pm$ 0·47 (10 measurements) on the transmittance scale with slit width of 2 mm at 460 mμ. Between each of these readings the cuvette was removed and replaced. K. A. PROCTOR

2058. **Cooling liquid samples for X-ray fluorescence analysis.** W. D. Moak (Atomic Power Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1906.—The specimen holder described by Tomaino and DePietro (*Norelco Reptr.*, 1956, **3**, 57) has been slightly modified, and a flow of supercooled helium over the sample is used to dissipate heat. K. A. PROCTOR

2059. **Generalised X-ray emission spectrographic calibration applicable to varying compositions and sample forms.** H. D. Burnham, J. Hower and L. C. Jones (Shell Oil Co., Wood River, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1827–1834.—Alterations and extensions to Sherman's equations allow X-ray emission spectroscopy to be applied quant. to ternary mixtures in samples of non-identical geometry, over a wide range of composition. The method has been applied to the determination of Cr, Fe and Ni in steel. K. A. PROCTOR

2060. **Electron probe X-ray micro-analyser.** L. S. Birks and E. J. Brooks (U.S. Naval Res. Lab., Washington). *Rev. Sci. Instrum.*, 1957, **28** (9), 709–712.—A 1 to 3-micron electron probe for X-ray spectrochemical analysis is described. By operating at 20 to 30 kV and at less than 0·1 μA beam current, counting rates at about 3000 counts per sec. are obtained from pure elements such as iron, thus permitting detection of concn. as low as a few tenths per cent. With increased voltage and current, and beam size 10 to 20 microns, counting rates may be increased to 50,000 per sec. G. SKIRROW

2061. **A direct-reading fluorimeter.** L. Brealey and R. E. Ross (Standards Dept., Boots Pure Drug Co. Ltd., Beeston, Nottingham). *Analyst*, 1957, **82**, 769–773.—The fluorimeter, the construction of which is described, is designed for use with large numbers of samples and is fitted with a run-through cuvette, enabling the samples to be run through in quick succession. An additional feature is that very dilute soln. subject to photo-decomposition may be run slowly through the cuvette so that decomposition is negligible. The instrument is highly sensitive, has good long-term and short-term stability, does not require too frequent re-calibration and is mains-operated. A. O. JONES

2062. **Conversion of refractive dispersions.** H. M. Eby and R. A. Klett (Universal Oil Prod. Co., Des Plaines, Ill., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 100–103.—Refractive dispersions obtained with mercury g and e lines can be converted to the equiv. hydrogen F and C line dispersions by the use of a correlation equation based on a conversion table. Data obtained with mercury light sources can thus be used for the estimation of aromatic hydrocarbon content. K. A. PROCTOR

Thermal

2063. **Accessories for the Chevenard thermobalance.** A. E. Newkirk (Gen. Elect. Res. Lab., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1958, **30** (1), 162.—The usefulness of the thermobalance can be increased by the addition of several easily constructed accessories such as a furnace liner (to facilitate cleaning) and a transparent top-plate so that a sample may be observed during heating. K. A. PROCTOR

2064. **Inexpensive automatic recording thermobalance.** W. W. Wendlandt (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chem.*, 1958, **30** (1), 56–58.—The construction and operation of the instrument which has been built from a torsion balance of 0 to 102-mg capacity are described. The apparent accuracy for a 100-mg sample is approx. 0·5% and the reproducibility is 0·20%, which compares favourably with that of commercial models. K. A. PROCTOR

2065. **An apparatus for the determination of total gas in fuel element samples.** R. I. Miller (Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. HW-51452, 1957, 13 pp.—The method described is limited to the measurement of total gases evolved by a uranium sample of specific dimensions, but six samples can be analysed in 8 hr. In the apparatus described, 8 samples are loaded into a tube at once and moved magnetically one by one into a furnace heated by induction. The pressure rise is automatically indicated by an Alphatron Vacuum Gauge which drives a strip chart recorder. N. E.

Electrical

2066. **Controlled potential electrolysis. I. Apparatus and general procedure.** Masao Tanaka (Bessi Mine Office, Sumitomo Metal Mining Co., Ltd., Niihama, Ehime Prefecture). *Japan Analyst*, 1957, **6** (6), 341–343.—The apparatus and analytical procedures are described for the controlled potential electrolysis of various metal ions in several supporting electrolytes. For electrolytic analysis, the cathode potential was kept more negative than the deposition potential (polarographically measured) by 0·2 V.

II. Electrolysis in hydrochloric acid solution. Masao Tanaka. *Ibid.*, 1957, **6** (6), 344–349.—The

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alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	$R_{\mathfrak{B}}$
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

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centimetre	cm
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concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
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microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
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molar (concentration)	M
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relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
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